Quality Systems Manual for Environmental Laboratories

DRAFT FOR REVIEW

Based On
National Environmental Laboratory Accreditation Program (NELAP)
Chapter 5 (Quality Systems)
NELAP Voted Version 12 – 1 July 1999

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PREFACE TO THE DoD QUALITY SYSTEMS MANUAL

Purpose

The purpose of this document is to provide implementation guidance on the establishment and management of quality systems for environmental testing laboratories that intend to perform work for DoD. This guidance is based ~~up~~on National Environmental Laboratory Accreditation Conference's (NELAC) Quality System requirements, and provides implementation clarification and expectations for DoD environmental programs. It is designed to serve as a standard reference for DoD representatives from all components who design, implement, and oversee contracts with environmental testing laboratories.

Background

To be accredited under the National Environmental Laboratory Accreditation Program (NELAP), laboratories shall have a comprehensive Quality System in place, the requirements for which are outlined in NELAP Chapter 5 (Quality Systems). Using NELAP Chapter 5 as its textual base, the "DoD Quality Systems Manual" is designed to replace common components of the following documents, previously issued by individual components of DoD:

- United States Navy - *Installation Restoration Laboratory Quality Assurance Guide*, Interim Document, February 1996.
- Air Force Center for Environmental Excellence - *Quality Assurance Project Plan, Version 3.* March 1998.
- Army Corps of Engineers (USACE – HTRW) – *Interim Chemical Data Quality Management (CDQM) Policy for USACE HTRW Projects.* 8 December 1998.

In combining the common components of these three documents, this Manual allows laboratories to design Quality Systems to meet basic requirements for laboratory accreditation under NELAP, as well as the implementation needs of all DoD components. The document achieves this by clarifying and elaborating upon DoD's expectations of the laboratory, with respect to the implementation of specific components of the NELAC Quality System.

Full implementation of this Manual's requirements is expected within two years following release. This standardized document is only one of several efforts planned for implementation by DoD. As such, until such time as further standardization by DoD occurs, this document may be supplemented by component-specific requirements. In addition, specific requirements outlined in project-specific QAPP's will also provide additional guidance that shall be followed. Requirements contained in this Manual are superceded by more stringent or more specific project-specific requirements or regulations. The laboratory bears the responsibility for meeting all State requirements. Nothing in this document relieves any laboratory from complying with contract requirements or with Federal, State, and/or local regulations.

Results and Benefits

The side-by-side integration of NELAP requirements with DoD implementation clarifications creates several benefits for the laboratory, DoD, and the regulatory communities.

- Standardization of Processes – Because this Manual provides laboratories with a comprehensive set of requirements that meet the needs of all DoD clients, as well as NELAP, the laboratory may use it to create a standardized Quality System. Ultimately, this standardization will save laboratory resources, by establishing one set of consistent requirements for all DoD environmental work. The standardized guidance will also serve to "level the playing field" for laboratories competing for DoD contracts, because the expectations will be identical across all DoD components. An audit that

satisfies the needs of one component will satisfy comparable needs of the other components as well. As such, this Manual will facilitate the standardization of audits, which are consistent and transferable between components. The result will be saved resources for both the government and private sector.

- **Deterrence of Fraud** – Fraudulent activities by only a few laboratories have implications throughout the industry, with negative impacts upon all laboratories. This Manual addresses this issue, establishing a minimum threshold program for all laboratories to use to deter and detect fraud.

- **Compliance Requirement Specification** – Because this Manual applies to all laboratories performing environmental work for DoD, it represents the first policy guidance for laboratories involved in compliance testing.

- **Foundations for the Future** – A standardized approach to Quality Systems, shared by laboratories, NELAP, and DoD paves the way for the standardization of other processes in the future. For example, this Manual might serve as a platform for a standardized strategy for Performance Based Measurement System (PBMS) implementation. In addition, as noted above, DoD plans to supplement this document with other standardized tools, including standard report formats.

Audience

This Manual is designed to meet the needs of the following audiences:

- Public (i.e., government) and private laboratories, contracted with DoD either directly, or through a prime contractor or subcontractor;
- DoD Implementing Agency representatives, who will use this document to ensure consistency with NELAP when drafting contracts; and
- DoD Oversight Personnel and Assessors, who will use this document to uniformly and consistently evaluate the laboratory's implementation of NELAP and DoD program requirements.

Document Format

Because the DoD Quality Systems Manual is designed to complement and implement NELAP Chapter 5 (Quality Systems), that document serves as the primary text for this Implementation Manual. The section numbering has been slightly changed from that of NELAP Chapter 5 as the manual is meant to be a stand-alone document. The number 5 has been eliminated from all section and sub-section headings. However, second level numbering has been retained to ensure maintenance of a parallel organization to the NELAC Quality Systems requirements. For instance, Section 5.4.2 in NELAP Chapter 5 (referencing Chapter 5 of the NELAC standards) is equivalent to Section 4.2 in this manual. In addition, there are two sets of appendices to this DoD manual. The first set is the NELAC appendices, modified with DoD Clarification Boxes. The second set is DoD appendices. The DoD appendices will include specific area of standardization focus that will be implemented across all DoD components for laboratory services. DoD clarifications that elaborate upon specific NELAP requirements are presented in gray text boxes, placed at the applicable section of the document. This allows laboratories preparing for NELAP accreditation to implement their Quality Systems in a way that fulfills the needs of DoD, as well as NELAP. For ease of reference, each gray box in the draft document is numbered.

ACROYNM LIST

°C: Degrees Celsius
ANSI/ASQC: American National Standards Institute/American Society for Quality Control
ASTM: American Society for Testing and Materials
CAS: Chemical Abstract Service
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
CLP: Contract Laboratory Program
COC: Chain-of-custody
CV: Coefficient of variation
DO: Dissolved oxygen
DOC: Demonstration of capability
DoD: Department of Defense
DQOs: Data quality objectives
EC: Exposure concentration
EPA: Environmental Protection Agency
g/L: Grams per liter
GC/MS: Gas chromatography/mass spectrometry
ICP-MS: Inductively coupled plasma-mass spectrometer
ICV: Initial calibration verification
ID: Identifier
ISO/IEC: International Standards Organization/International Electrotechnical Commission
LC50: Lethal concentration at 50%
LCS: Laboratory control sample
LQMP: Laboratory Quality Management Plan
MDL: Method detection limit
mg/kg: Milligrams per kilogram
MQO: Measurement quality objective
MS: Matrix spike
MSD: Matrix spike duplicate
MSD: Minimum significant difference
NELAC: National Environmental Laboratory Accreditation Conference
NELAP: National Environmental Laboratory Accreditation Program
NIST: National Institute of Standards and Technology
NOEC: No-observable-effects concentration
OSHA: Occupational Safety and Health Administration
PBMS: Performance-Based Measurement System
PC: Personal computer
PCBs: Polychlorinated biphenyls
PT: Proficiency testing
QA: Quality assurance
QAD: Quality Assurance Division (EPA)
QAMS: Quality Assurance Management Section
QAPP: Quality Assurance Project Plan
QC: Quality control
RL: Reporting limit
RPD: Relative percent difference
RSD: Relative standard deviation
SD: Serial dilutions
SOP: Standard operating procedure
TAC: Test Acceptability Criteria
TSS: Total suspended solids
UV: Ultraviolet
VOC: Volatile organic compound
WET: Whole effluent toxicity

QUALITY SYSTEMS

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a Quality Manual and followed to ensure and document the quality of the analytical data. Laboratories seeking accreditation under the National Environmental Accreditation Program (NELAP) must assure implementation of all QA policies and the essential applicable QC procedures specified in this chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

The intent of this Chapter is to provide sufficient detail concerning quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

NELAC is committed to the use of Performance Based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter 5 is organized according to the structure of ISO/IEC Guide 25, 1990. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC Guide 25.

All items identified in this chapter shall be available for on-site inspection or data audit.

1.0 SCOPE

a) This Standard sets out the general requirements in accordance with which a laboratory has to demonstrate that it operates, if it is to be recognized as competent to carry out specific environmental tests.

b) This Standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed.

c) This Standard is for use by environmental testing laboratories in the development and implementation of their quality systems. It shall be used by accreditation authorities, in assessing the competence of environmental laboratories.

Scope of DoD Document:

- These standards are applicable to any laboratory providing sample analysis to support environmental programs for DoD installations and facilities within the United States and its possessions.
- These standards are intended to apply to laboratories that produce definitive data, regardless of the methods being applied (i.e., technically defensible and legally admissible data).
- These standards may be supplemented by project-specific requirements, as agreed upon by the agency, regulators, laboratories, and other involved parties.
- The laboratory bears the responsibility for meeting all State requirements. Nothing in this document relieves any laboratory from complying with contract requirements or with Federal, State, and/or local regulations.

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2.0 REFERENCES

See Appendix A.

3.0 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ISO 8402, ANSI/ASQC E-4, 1994, the EPA "Glossary of Quality Assurance Terms and Acronyms," and the *International vocabulary of basic and general terms in metrology (VIM*) are applicable, the most relevant being quoted in NELAP Chapter 1 Appendix A Glossary together with further definitions applicable for the purposes of this Standard.

Definitions: For reference purposes, applicable terms from the NELAC Glossary are included as Appendix B in this DoD Manual. Furthermore, additional terms not currently included in the NELAP Glossary are defined by DoD to aid the laboratory in implementing this standard appropriately. These terms are also in Appendix B.

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4.0 ORGANIZATION AND MANAGEMENT

4.1 Legal Definition of Laboratory

The laboratory shall be legally identifiable. It shall be organized and shall operate in such a way that its permanent, temporary, and mobile facilities meet the requirements of this Standard**.**

4.2 Organization

The laboratory shall:

a) Have managerial staff with the authority and resources needed to discharge their duties;

b) Have processes to ensure that its personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of their work;

c) Be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;

d) Specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform, or verify work affecting the quality of calibrations and tests;

   Such documentation shall include:

   1) A clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured and

   2) Job descriptions for all positions.

e) Provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test, and the assessment of the results. The ratio of supervisory to nonsupervisory personnel shall be such as to ensure adequate supervision, to ensure adherence to laboratory procedures and accepted techniques.

f) Have a technical director(s) (however named) who has overall responsibility for the technical operation of the environmental testing laboratory.

The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

The technical director(s) shall meet the requirements specified in the Accreditation Process. (See NELAC Section 4.1.1.1.)

g) Have a quality assurance officer (however named) who has responsibility for the quality system and its implementation. The quality assurance officer shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical director. Where staffing is limited, the quality assurance officer may also be the technical director or deputy technical director.

The quality assurance officer (and/or his/her designees) shall:

1) Serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) Have functions independent from laboratory operations for which they have QA oversight;

3) Be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) Have documented training and/or experience in QA/QC procedures and be knowledgeable in the Quality System, as defined under NELAC;

5) Have a general knowledge of the analytical test methods for which data review is performed;

6) Arrange for or conduct internal audits on the entire technical operation annually; and

7) Notify laboratory management of deficiencies in the quality system and monitor corrective action.

Quality Assurance – Duty of Quality Assurance Officer: The Quality Assurance Officer shall also be responsible for ensuring continuous improvement at the laboratory through the use of control charts and other method performance indicators (e.g., PT samples and internal and external audits).

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h) Nominate deputies in case of absence of the technical director(s) and/or quality assurance officer;

i) Have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories);

j) When available, participate in inter-laboratory comparisons and proficiency testing programs. For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in NELAP Chapter 2.0.

Technical Directors – Responsibility of Technical Directors: ~~Technical directors are~~ Lab management is responsible for following through with proficiency testing programs and for ensuring that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions.

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5.0 QUALITY SYSTEM – ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS, AND DATA VERIFICATION

5.1 Establishment

The laboratory shall establish and maintain a quality system based on the required elements contained in this Chapter and appropriate to the type, range, and volume of environmental testing activities it undertakes.

a) The elements of this Quality System shall be documented in the organization's quality manual.

b) The quality documentation shall be available for use by the laboratory personnel.

Quality System Documentation: This documentation includes the Quality Manual, Standard Operation Procedure (SOP) documents, and other appropriate reference documents and texts.

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c) The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

d) The laboratory management shall ensure that these policies and objectives are documented in a Quality Manual and communicated to, understood, and implemented by all laboratory personnel concerned.

e) The Quality Manual shall be maintained current under the responsibility of the quality assurance officer.

5.2 Quality Manual

The Quality Manual and related quality documentation shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard.

The Quality Manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units, which are to be covered by this quality manual; and the effective date of the version.

Quality Manual Updating: The following list reflects topic areas that shall be included in the Quality Manual. Additional details about each topic area are provided in the sections that follow. The Manual shall be reviewed at least annually for accuracy and adequacy, and updated as appropriate. All such reviews shall be documented and available for inspection.

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The Quality Manual and related quality documentation shall also contain:

a) A quality policy statement, including objectives and commitments, by top management;

b) The organization and management structure of the laboratory, its place in any parent organization, and relevant organizational charts;

Corporations – Laboratory Relationships ~~W~~w~~ith~~ Corporations: This includes the laboratory's relationship(s) to corporate affiliations and networks.

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c) The relationship between management, technical operations, support services, and the quality system;

d) Procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system that ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

Document Control – Distribution: Consistent with the definition of "Document Control" provided in NELAP Appendix B, this control system shall ensure ~~that each updated SOP is distributed to~~ all analysts implementing the task(s) or procedure(s) described in that SOP shall be made individually aware that changes to an SOP have occurred. A copy of the updated SOP shall be available in close proximity to the work station (i.e., within the same work area).

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e) Job descriptions of key staff and reference to the job descriptions of other staff;

Personnel To Be Included in Quality Manual: At a minimum, the following managerial and supervisory staff (however named), shall be considered key staff, and their job descriptions included in the Quality Manual and other related documents:

(1) ~~(1)~~ Executive Staff (e.g., Chief Executive Officer, Chief Operating Officer, Laboratory Director, Technical Director);
(2) ~~(2)~~ Technical Directors/Supervisors (e.g., Section Supervisors for Organics and Inorganics);
(3) ~~(3)~~ Quality Assurance Systems Directors/Supervisors (e.g., QA Officer, Quality Auditors); and
(4) ~~(4)~~ Support Systems Directors/Supervisors (e.g., Information Systems Supervisor, Purchasing Director, and Project Managers). In addition, the Quality Manual shall include job descriptions for key staff in each of these four areas, as appropriate to the laboratory.

If the size and organization of the laboratory precludes separate managers and/or supervisors in each of these key areas, the functions covered in the four areas shall be addressed in the job descriptions provided for the key staff.

~~Finally, t~~The Quality Manual shall describe the relationship of the key staff listed above to other technical and support staff. Any changes in key personnel for the laboratory must be documented to all laboratory users.

Technical staff ~~are~~is those individuals who conduct the work of the laboratory (e.g., sample receipt and documentation staff, the chemists who run the analytical equipment). Support staff administers the business practices of the laboratory, as well as information management and contractual systems. Quality Assurance staff oversees the implementation of the quality system, and reports to the Quality Assurance Officer or his/her designee.

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f) Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the QA officer(s), technical director(s), and the agent who is in charge of all laboratory activities, such as the laboratory director or laboratory manager;

g) The laboratory's procedures for achieving traceability of measurements;

Traceability of Measurements: Standards addressing this issue are included in Section 9.0 (Measurement Traceability and Calibration), Section 10.5 (Documentation and Labeling of Standards and Reagents), and Section 12.0 (Records).

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h) A list of all test methods under which the laboratory performs its accredited testing;

i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) Reference to the calibration and/or verification test procedures used;

k) Procedures for handling submitted samples;

l) Reference to the major equipment and reference measurement standards used, as well as the facilities and services used by the laboratory in conducting tests;

m) Reference to procedures for calibration, verification, and maintenance of equipment;

n) Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials, and internal quality control schemes;

o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected or departures from documented policies and procedures occur;

p) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) Procedures for dealing with complaints;

r) Procedures for protecting confidentiality (including national security concerns) and proprietary rights;

s) Procedures for audits and data review;

Audits – Quality Manual Specification: The Quality Manual shall also specify which records are considered necessary to conduct an adequate review.

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t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

u) Processes/procedures for educating and training personnel in their ethical and legal responsibilities, including the potential punishments and penalties for improper, unethical, or illegal actions;

Personnel Training – Ethical: Additional descriptions related to this requirement are included in Section 6.2.

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v) Reference to procedures for reporting analytical results; and

w) A Table of Contents and applicable lists of references, glossaries, and appendices.

5.3 Audits

Audits – Section Summary: The following subsections of 5.3 refer to Internal Assessment Tools to be used by the laboratory. Section 5.3.1 discusses Systems and Technical Audits, both of which shall be conducted annually to evaluate whether the quality system is being implemented at the operational level of the laboratory. Section 5.3.2 addresses higher-level managerial reviews, designed to evaluate whether the quality system itself is effective. These can be done in conjunction with each other or separately, at the discretion of the laboratory. This section also addresses requirements for a Fraud Prevention program. Section 5.3.3 addresses the review of all auditing activities. Section 5.3.4 addresses continuous quality control practices, that shall be conducted by the laboratory on an ongoing basis.

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5.3.1 Internal Audits

The laboratory shall arrange for annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's quality system. It is the responsibility of the quality assurance officer to plan and organize audits as required by a predetermined schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, whenever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.

Audits – Internal: These Internal Audits shall include both Technical and Systems Audits. They may be scheduled or unannounced. Technical Audits verify compliance with method-specific requirements, as well as operations related to the test method (e.g., sample preparation). (These operations include all actions related to data generation and the assurance of its quality.) Systems Audits verify compliance with the laboratory's quality system, based upon the NELAP Quality System, and documented in the laboratory's Quality Manual. Response to complaints, sample acceptance policies, and sample tracking methodologies are examples of procedures that would be reviewed as part of a Systems Audit. Data Audits are considered a subset of Technical Audits.

An audit schedule shall be established such that all elements/areas of the laboratory are reviewed over the course of one year.

Personnel performing an internal audit shall complete the audit under the direction of the Quality Assurance Officer, however named. To be considered "trained and qualified," the Internal Auditor shall be trained and qualified in conducting the type of audit under review.

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5.3.2 Managerial Review

The laboratory management shall conduct a review, at least annually, of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors. The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

Audits – Managerial Review: This is a separate review from the Internal Audit discussed in Section 5.3.1, and shall be completed by laboratory managerial personnel. As noted in clarification box number 13, however, Internal Audits and Managerial Reviews may be conducted in conjunction with each other.

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5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory management shall ensure that these actions are discharged within the agreed timeframe.

Audits – Timeframe of Audit Review: The timeframe for these actions shall be based upon the magnitude of the problem and its impact upon the defensibility and use of data.

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5.3.4 Performance Audits

In addition to periodic audits, the laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of such checks are:

a) Internal quality control procedures using, whenever possible, statistical techniques (See Section 5.4 below);

b) Participation in proficiency testing or other interlaboratory comparisons (See NELAC Chapter 2.0);

c) Use of certified reference materials and/or in-house quality control using secondary reference materials, as specified in Section 5.4;

d) Replicate testings using the same or different test methods;

e) Re-testing of retained samples; and

f) Correlation of results for different parameters of a sample (e.g., total phosphorus should be greater than or equal to orthophosphate).

Audits – Laboratory Checks of Performance Audits: This section requires the laboratory to continuously evaluate the quality of generated data, by systematically and routinely implementing control checks that go beyond those required by the test methods. The results of these checks (examples of which are listed above) shall be routinely reviewed after they are performed to monitor and evaluate the quality and usability of data generated by the laboratory. Although a supplemental review of these checks shall be included as part of the annual internal audits, the laboratory shall also ensure that the results of these checks are reviewed (and corrective action taken) on a regular and timely basis following the actual completion of the check to remedy the problem, avoid its reoccurrence, and improve the Quality System overall.

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5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (Section 10.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures, and quality control have occurred. These procedures shall include, but are not limited to, the following:

1) Identify the individual(s) responsible for assessing each QC data type;

2) Identify the individual(s) responsible for initiating and/or recommending corrective actions;

3) Define how the analyst should treat a data set if the associated QC measurements are unacceptable;

4) Specify how out-of-control situations and subsequent corrective actions are to be documented; and

5) Specify procedures for management (including the QA officer) to review corrective action reports.

Audits – Corrective Action: Managersment, including the QA Officer, isare also responsible for acting upon these reviews corrective action report reviews, ensuring that corrective actions are taken, and checking the adequacy of those corrective actions. Furthermore, managersment isare ultimately accountable for the follow-through, and verification and evaluation of these corrective actions. Further explanatory clarifications of DoD expectations are provided as follows:

Nonconformance. The laboratory shall have an established, documented policy and procedures to identify and control work and test results that do not or may not meet expected or specified requirements, or are nonconforming or suspected to be nonconforming. Policy and procedures shall ensure that:

- Responsibilities and authorities for the managingement of nonconforming work/results are designated.
- Actions to be taken upon identification of a nonconformance are defined and implemented, and include, but are not limited to: evaluating the significance of a nonconformance; halting work and investigating the contributors to the nonconformance (e.g., equipment, personnel, methods); withholding of reports and certificates, as necessary; informing clients of nonconformance resulting from their samples and the need to recall results of nonconforming work already released to them; and implementing corrective action as needed and evaluating the results. (See corrective action requirements below.)

Corrective Action. The laboratory shall have an established, documented policy, and procedures for actions to be taken to eliminate the causes of a nonconformance and to prevent recurrence. The corrective action process shall identify and implement corrective actions likely to eliminate the root cause of nonconformance(s). Laboratory policies and procedures shall ensure that:

- Responsibilities and authorities for instituting corrective action are designated.
- Possible causes of the nonconformance(s) are investigated.
- Root cause analysis is performed.
- Changes resulting from corrective action are recorded and retrievable.
- Corrective action(s) are monitored.
- Preventative action is taken to prevent recurrence.

Monitoring of Corrective Actions. After implementation of corrective action(s), the laboratory shall monitor their effect to determine if action(s) taken are effective in overcoming the nonconformance identified (i.e., the root cause has been eliminated and its reoccurrence prevented). Historical corrective action reports should be periodically reviewed to identify long-term trends or recurring problems.

Preventive Action. All operations shall be systematically and thoroughly reviewed at regular intervals to:

- Obtain input on the laboratory's operations;
- Determine what considerations need to be given to input (from reviews); and
- Determine how corrective action(s), if necessary, shall be carried out.

Reference: American Society for Quality Control. 1991. *Q2 – Quality Management and Quality System Elements for Laboratories – Guidelines.*

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b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, all samples

associated with the failed quality control measure shall be reported with the appropriate data qualifier(s).

Data Qualifiers: ~~These standard data qualifiers include the following:~~ Some of the standard data qualifiers, to be used by laboratories only, are listed below. Additional data qualifiers (i.e., R – Rejected) may be used by data validators when evaluating data usability.

U – Undetected; The associated number is the ~~method~~ reporting limit, adjusted by any dilution factor used in the analysis.
J – Estimated; The analyte was positively identified; the quantitation is an estimation (e.g., matrix interference, below standard, outside the calibration range).
B – Blank contamination; Analyte detected above the ~~method~~ reporting limit in an associated blank.
~~R – Rejected; Data are unusable for their intended project use.~~
N – Nontarget analyte; Tentatively identified compound (using mass spectroscopy).
Q – One or more quality control criteria (e.g., calibration, LCS recovery, surrogate spike recovery, etc.) failed. Data usability should be carefully assessed.

When other flags are required contractually (e.g., CLP), these may be substituted, as appropriate.

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5.4 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall ensure that the applicable principles are addressed:

a) All laboratories shall have protocols in place to monitor the following quality controls:

Quality Control Actions: Quality control actions should be both batch-specific and time-based (i.e., those required to be conducted at specific time periods, such as for tunes and method detection limits [MDLs]). Batch- specific quality control actions include sample-specific quality control actions such as surrogate spikes.

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1) Adequate positive and negative controls to monitor tests, such as blanks, spikes, reference toxicants;

2) Adequate tests to define the variability and/or repeatability of the laboratory results, such as replicates;

3) Measures to assure the accuracy of the test method, including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability, such as linearity;

5) Selection of appropriate formulae to reduce raw data to final results, such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

6) Selection and use of reagents and standards of appropriate quality;

~~6)~~ 7) Measures to ensure the selectivity of the test for its intended purpose; and

~~7~~8) Measures to ensure constant and consistent test conditions (both instrumental and environmental) where required by the test method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an ongoing basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D.)

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See Section 11.2, Sample Acceptance Policy.)

d) The quality control protocols specified by the laboratory's method manual (Section 10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into its method manuals.

The essential quality control measures for testing are found in Appendix D of this chapter.

6.0 PERSONNEL

6.1 General Requirements for Laboratory Staff

The laboratory shall have sufficient personnel, having the necessary education, training, technical knowledge, and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of his/her particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures, and records management.

Technical Directors - Qualifications: Required qualifications for the Technical Director(s) are addressed further below. DoD stresses that a director or designee meeting the qualifications below shall be present in each area of analytical service. Laboratory management, as addressed in Section 6.2, is defined as designees (e.g., Laboratory Manager, Technical Director, Supervisors, and Quality Assurance Officers, however named) having oversight authority and responsibility for laboratory output.

The following requirements are direct excerpts from **NELAP Chapter 4 (Accreditation Process), Revision 12 – July 1, 1999**.

4.1.1 Personnel Qualifications

Persons who do not meet the education credential requirements of Section 4.1.1.1 of the NELAC standards and are the technical director(s) on the date that the laboratory becomes subject to these NELAC Standards, and obtain~~s~~ accreditation, shall qualify as technical director(s) for the field of testing of that laboratory or any other NELAC-accredited laboratory.

4.1.1.1 Definition, Technical Director(s)

The technical director(s) means a full-time member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory procedures and reporting of results. The title of such person may include, but is not limited to, laboratory director, technical director, laboratory supervisor, or laboratory manager. A laboratory may appoint one or more technical directors for the appropriate fields of testing for which they are seeking accreditation. His/her name shall appear in the national database. This person's duties shall include, but not be limited to, monitoring standards of performance in quality control and quality assurance; monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data; ensuring that sufficient numbers of qualified

personnel are employed to supervise and perform the work of the laboratory; and providing educational direction to laboratory staff. An individual shall not be the technical director(s) of more than one accredited environmental laboratory without authorization from the primary Accrediting Authority. Circumstances to be considered in the decision to grant such authorization shall include, but not be limited to, the extent to which operating hours of the laboratories to be directed overlap, adequacy of supervision in each laboratory, and the availability of environmental laboratory services in the area served. The technical director(s) who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director(s) to temporarily perform this function. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

Qualification of the Technical Director(s):

a) Any technical director of an accredited environmental laboratory engaged in chemical analysis shall be a person with a bachelors degree in the chemical, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

b) Any technical director of an accredited environmental laboratory limited to inorganic chemical analysis, other than metals analysis, shall be a person with at least an earned associate's degree in the chemical, physical, or environmental sciences, or two years of equivalent and successful college education, with a minimum of 16 college semester credit hours in chemistry. In addition, such a person shall have at least two years of experience performing such analysis.

c) The technical director(s) of an accredited environmental laboratory engaged in microbiological or biological analysis shall be a person with a bachelors degree in microbiology, biology, chemistry, environmental sciences, physical sciences, or engineering with a minimum of 16 college semester credit hours in general microbiology and biology and at least two years of experience in the environmental analysis of representative analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

   A person with an associate's degree in an appropriate field of the sciences or applied sciences, with a minimum of four college semester credit hours in general microbiology may be the technical director(s) of a laboratory engaged in microbiological analysis limited to fecal coliform, total coliform, and standard plate count. Two years of equivalent and successful college education, including the microbiology requirement, may be substituted for the associate's degree. In addition, each person shall have one year of experience in environmental analysis.

d) Any technical director of an accredited environmental laboratory engaged in radiological analysis shall be a person with a bachelor's degree in chemistry, physics, or engineering with 24 college semester credit hours of chemistry with two or more years of experience in the radiological analysis of environmental samples. A master's or doctoral degree in one of the above disciplines may be substituted for one year experience.

e) Any technical director of an accredited environmental laboratory engaged in microscopic examination of asbestos and/or airborne fibers shall meet the following requirements:

   i) For procedures requiring the use of a transmission electron microscope, a ~~bachelors~~bachelor's degree, successful completion of courses in the use of the instrument, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

ii) For procedures requiring the use of a polarized light microscope, an associate's degree or two years of college study, successful completion of formal coursework in polarized light microscopy, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

iii) For procedures requiring the use of a phase contrast microscope, as in the determination of airborne fibers, an associate's degree or two years of college study, documentation of successful completion of formal coursework in phase contrast microscopy, and one year of experience, under supervision, in the use of the instrument.

f) Any technical director of an accredited environmental laboratory engaged in the examination of radon in air shall have at least an associate's degree or two years of college and one year of experience in radiation measurements, including at least one year of experience in the measurement of radon and/or radon progeny.

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6.2 Laboratory Management Responsibilities

In addition to Section 4.2.d., the laboratory management shall be responsible for:

a) Defining the minimal level of qualification, experience, and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills, such as using a balance, colony counting, aseptic, or quantitative techniques, shall be considered.

b) Ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration shall be documented (See Appendix C).

Note: In laboratories with specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented).

Work Cell – Definition of Work Cell: Additional guidance on this issue is provided in Section 10.2.1.f and g. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation/extraction and analysis. To ensure that the entire preparation-extraction-analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the Demonstration of Capability at each individual step in the sequence as performed by each individual analyst/team member, remains of utmost importance. A work cell may NOT be defined as a group of analysts that performs the same step in the same process (e.g., extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

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c) Ensuring that the training of each member of its technical staff is kept up-to-date (on-going) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques, or laboratory procedures shall all be documented.

3) Training courses in legal and ethical responsibilities include the potential punishments and penalties for improper, unethical, or illegal actions. Evidence must also be on file that

demonstrates that each employee has read, acknowledged, and understood their personal ethical and legal responsibilities, including the potential punishments and penalties for improper, unethical or illegal actions.

Personnel Training – Ongoing: Additional descriptions related to this requirement are included in Section 6.2.h

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4) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood, and agreed to perform the most recent version of the test method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

   i. Acceptable performance of a blind sample (single blind to the analyst);

   ii. Another demonstration of capability;

   iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., gas chromatography/mass spectrometry [GC/MS] volatiles by purge and trap for 524.2, 624, or 5035/8260 would only require documentation for one of the test methods);

   iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

   v. If i-iv cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results.

d) Documenting all analytical and operational activities of the laboratory.

e) Supervising all personnel employed by the laboratory.

f) Ensuring that all sample acceptance criteria (Section 11.0) are verified and that samples are logged into the sample tracking system and properly labeled and stored.

g) Documenting the quality of all data reported by the laboratory.

h) Developing a proactive program for the prevention and detection of improper, unethical, or illegal actions. The components of this program could include: internal Proficiency testing (single and double blind); post-analysis electronic and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.

~~Fraud~~A Program to Detect and Prevent Improper, Unethical, or Illegal Actions ~~Prevention Program~~: In order to perform work for DoD under this Manual, the laboratory shall have a documented program to prevent ~~Fraud~~improper, unethical, or illegal actions ~~Prevention Program~~. To facilitate the implementation of this required program, DoD has compiled the following text to (1) clearly define ~~the term fraud~~improper, unethical, or illegal actions, (2) outline ~~fraud~~improper, unethical, or illegal actions prevention and detection program elements, and (3) identify examples of inappropriate (i.e., potentially fraudulent) laboratory practices. Data shall be produced according to the project-specific requirements as specified in the final approved project documents, such as the approved QAPP. The laboratory shall be aware of these requirements and be able to show that these requirements were followed.

Definition. ~~Laboratory fraud is defined~~Improper actions are defined as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional. Unethical or

illegal actions are defined as the deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable. ~~It is also defined as an intentional gross deviation from contract-specified or method-specified analytical practices, combined with the intent to conceal the deviation.~~ Prevention of laboratory ~~fraud~~improper, unethical, or illegal actions begins with a zero tolerance philosophy established by management. ~~Fraud is~~Improper, unethical, or illegal actions are detected through the implementation of oversight protocols.

~~Fraud~~Prevention and Detection Program for Improper, Unethical, or Illegal Actions ~~Detection & Prevention Program~~. Laboratory management shall implement a variety of proactive measures to promote prevention and detection of ~~fraudulent~~ improper, unethical, or illegal activities. The following components constitute the baseline and minimum requirements for a ~~fraud~~improper, unethical, or illegal actions prevention program and shall be included as part of the laboratory's comprehensive quality program:

- An ethics policy that is read and signed by all personnel;
- Initial and annual ethics training;
- Internal audits, as described elsewhere in Section 5.3;
- Inclusion of anti-fraud language in subcontracts;
- Analyst notation and sign-off on manual integration changes to data (See also ~~Section 8.a~~DoD Clarification Boxes #28 and #36); and
- Active use of electronic audit functions are mandatory, when they are available in the instrument software (see also Section 12.0); and
- A "no-fault" policy that encourages laboratory personnel to come forward and report fraudulent activities.

A proactive, "beyond the basics" approach to ~~fraud~~the prevention of improper, unethical, or illegal actions ~~prevention~~ is a necessary part of laboratory management. As such, in addition to the mandatory requirements above, the laboratory shall institute other ~~fraud~~actions to deter and detect improper, unethical, or illegal actions ~~deterrence and detection programs~~, as required by NELAC Section 6.2.4(h) (i.e., designate an ombudsman (data integrity officer) to whom laboratory personnel can report ~~potentially fraudulent~~improper, unethical, or illegal practices or provide routine communication of training, lectures, and changes in policy intended to reduce ~~fraud~~improper, unethical, or illegal actions).

Examples of ~~Data Fraud/Inappropriate~~Improper, Unethical, or Illegal Practices. Documentation that clearly shows how all analytical values were obtained shall be maintained by the laboratory, and supplied to the data user when necessary. To avoid miscommunication, a laboratory shall clearly document all errors, mistakes, and basis for manual integrations within the case narrative. Notification should also be made to the appropriate people such that appropriate corrective actions can be initiated. Gross deviations from specified procedures should be investigated for potential ~~fraud~~improper, unethical, or illegal actions, and findings of fraud prosecuted to the fullest extent of the law. Examples of ~~fraudulent~~improper, unethical, or illegal practices are identified below:

- ~~Inappropriate~~Improper use of manual integrations to meet calibration or method QC criteria ~~would be considered fraud~~ (e.g., peak shaving or peak enhancement are considered ~~fraudulent activities~~improper, unethical, or illegal actions if performed solely to meet QC requirements);
- Intentionally misrepresenting the date or time of analysis (e.g., intentionally resetting a computer system's or instrument's date and/or time to make it appear that a time/date requirement was met);
- ~~Manipulation of time travel of analyses to meet method 12-hour clock requirements;~~
- Falsification of results to meet method requirements;
- Reporting of results without analyses to support (~~e.g.~~i.e., dry-labbing);
- Selective exclusion of data to meet QC criteria (~~i.~~e.g., initial calibration points dropped without technical or statistical justification).

- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within LQMP that are not indicative of historical laboratory performance or used for batch control; and
- Notation of matrix inference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g., method blanks or laboratory control samples).
- Unwarranted manipulation of computer software (e.g., improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations).
- Improper alteration of analytical conditions (e.g., modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis.
- Misrepresentation of QC samples (e.g., adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over-spiking or under-spiking).
- Reporting results from the analysis of one sample for those of another.

References:

California Military Environmental Coordination Committee (EPA, CAL EPA, and DoD). March 1997. "Best Practices for the Detection and Deterrence of Laboratory Fraud."
Army Corps of Engineers (USACE – HTRW) – *Interim Chemical Data Quality Management (CDQM) Policy for USACE HTRW Projects.* 8 December 1998.

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6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory, including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in Section 10.2.1 for chemical testing. (See Section 6.2.c.)

7.0 PHYSICAL FACILITIES – ACCOMMODATION AND ENVIRONMENT

7.1 Environment

a) Laboratory accommodation, test areas, energy sources, lighting, heating, and ventilation shall be such as to facilitate proper performance of tests.

Environment–Cooling: Laboratory accommodation, test areas, energy sources, lighting, heating, cooling, and ventilation shall be such as to facilitate proper performance tests.

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b) The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.

c) The laboratory shall provide for the effective monitoring, control, and recording of environmental conditions, as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels.

d) In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

NOTE: It is the laboratory's responsibility to comply with the relevant health and safety requirements. This aspect, however, is outside the scope of this Standard.

7.2 Work Areas

a) There shall be effective separation between neighboring areas when the activities therein are incompatible, including culture handling or incubation areas and volatile organic chemicals handling areas.

b) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

c) Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

d) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) Access and entryways to the laboratory;

2) Sample receipt area(s);

3) Sample storage area(s);

4) Chemical and waste storage area(s); and

5) Data handling and storage area(s).

8.0 EQUIPMENT AND REFERENCE MATERIALS

Equipment Standards: Equipment shall be capable of achieving the accuracy, ~~and~~ precision, sensitivity, and selectivity required for the intended use of the generated data. The laboratory shall implement documented procedures to ensure that set-up, maintenance, and adjustments to instrument operating parameters are documented, and that adjustments to instruments do not exceed the limits specified in the approved SOPs.

The use of Outside Support Services and Supplies is further addressed in Section 15.0.

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a) The laboratory shall be furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is sought. In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the relevant requirements of this Standard are met.

b) All equipment shall be properly maintained, inspected, and cleaned. Maintenance procedures shall be documented.

c) Any item of the equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified, and, wherever possible, stored at a specified place until it has been repaired and shown by calibration, verification, or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

d) Each item of equipment, including reference materials, shall, when appropriate, be labeled, marked, or otherwise identified to indicate its calibration status.

e) Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed. These records shall include documentation on all routine and nonroutine maintenance activities and reference material verifications.

   The records shall include:

   1) The name of the item of equipment;

   2) The manufacturer's name, type identification, and serial number or other unique identification;

   3) Date received and date placed in service (if available);

   4) Current location, where appropriate;

   5) If available, condition when received (e.g., new, used, reconditioned);

   6) Copy of the manufacturer's instructions, where available;

   7) Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

   8) Details of maintenance carried out to date and planned for the future; and

   9) Histories of any damage, malfunction, modification, or repair.

9.0 MEASUREMENT TRACEABILITY AND CALIBRATION

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis. The laboratory shall have an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers, and control standards.

9.2 Traceability of Calibration

a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that, wherever applicable, measurements made by the laboratory are traceable to national standards of measurement, where available.

b) Calibration certificates, when available, shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.

c) Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results (e.g., by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis).

9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards have not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide, where possible, traceability to a national standard of measurement.

b) There shall be a program of calibration and verification for reference standards.

c) Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. Reference materials shall, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.

9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and (2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All support equipment shall be:

a) Maintained in proper working order. The records of all repair and maintenance activities, including service calls, shall be kept.

b) Calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

1) The equipment shall be removed from service until repaired; or

2) The laboratory shall maintain records of established correction factors to correct all measurements.

c) Raw data records shall be retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, incubators, and water baths shall be checked with NIST traceable references (where possible) in the expected use range. Additional monitoring as prescribed by the test method shall be performed for any device that is used in a critical test (such as incubators or water baths). The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on a monthly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

~~d)~~ f) For chemical tests, the temperature, cycle time and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

Autoclaves: The use of autoclaves during chemical tests is not typical, but is an analytical option for limited methods (e.g., mercury soil digestion). The typical use would be for sterilization purposes as described in item g below.

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~~d)~~ g) For biological tests, the sterilization temperature, cycle time, sterilization time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate by color change that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle. Demonstration of sterilization shall be provided by a continuous temperature recording or with the frequent use of spore strips.

Calibration – Calibration and Measurement Guidance: The following table provides specific guidance with respect to the calibration and performance measurements associated with specific types of analytical support equipment. The criteria presented that go beyond those established by the American Society for Testing and Methods (ASTM) Standards are currently in use by DoD Components. They are presented here in consolidated form, and will be formally adopted across DoD as a standardized requirement. ASTM Standards presented here are based upon the latest edition available at this Manual's publication date. As new editions are released, the latest revision of each ASTM Standard reference shall be followed, unless State or project requirements differ.

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Analytical Support Equipment Assessment	Frequency of Check	Acceptance Criteria	Calibration Check Procedures and Performance Criteria References (latest edition)
Balance calibration check	Daily or before use with two weights that bracket target weight(s) AND Annual calibration by certified technician	1% performance criterion to top-loading balances, and 0.1% to analytical balances. (Expanded criteria from 0.1 to 1% for top-loaders, for no standard existed for this balance type.)	ASTM E 898, Standard Practice for the Evaluation of Single-Pan Mechanical Balances, E 319, Standard Practice for the Evaluation of Single-Pan Mechanical Balances, and D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid
Refrigerator/Freezer temperature Monitoring	Daily	Refrigerators: 4 ± 2 °C, Freezers: -10 to -20° C (This ASTM standard does not address freezers, but SW-846 has noted this freezer range in some methods)	ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid
Thermometer calibration check	Mercury - annually Electronic - quarterly at two temperatures that bracket target temperature(s) against an NIST traceable thermometer	Appropriate correction factors applied	ASTM Methods E 77, Standard Test Method for Inspection and Verification of Thermometers, and D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid
~~Variable v~~Volumetric pipettes (fixed or variable) (~~i.e.~~e.g., Eppendorf)	Monthly	3% of known or true value. (Standard criteria for Class B transfer pipettes were used – tolerance varied depending on volume delivered, with widest % associated with smaller volume pipettes - 2.4% tolerance applied to 0.5 milliliter pipette – so expanded to 3% for consistency)	ASTM E 542, Standard Practice for Calibration of Volumetric Apparatus, and E 969, Standard Specification for Volumetric (Transfer) Pipettes
Nonvolumetric glassware/labware verification (Requirement applicable only when used for measuring initial sample and final extract/digestate volumes)	By lot at the time of purchase	3% of known or true value. (Standard tolerance does not exist – Class B volumetric flasks criteria vary between 0.8 to 0.05% for 5 mL to 2,000 mL, respectively – set at 3% to maintain consistency with pipette tolerance designation)	ASTM E 542, Standard Practice for Calibration of Volumetric Ware
Drying ovens	Before and after use	Compliance with method-specific requirements	ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid

9.4.2 Instrument Calibration

This standard specifies the essential elements that will define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data will be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

Calibration – Instrument: The DoD Implementation Clarifications included in Section 9.4.2 will each specify whether they are only applicable when method-specific guidance does not exist (e.g., when Performance Based Measurement System (PBMS) is being used) or whether they are applicable to all methods.

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9.4.2.1 *Initial Instrument Calibrations:*

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures, including calculations, integrations, and associated statistics must be included or referenced in the test method SOP.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

Calibration (Initial) – Raw Data Records: Raw records shall also include the analyst's name.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, dat~~a~~e, and signature/initials of person performing manual operation.

Applicable to all methods.

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c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

d) All initial instrument calibrations must be verified with a standard obtained from a second source and traceable to a national standard, when available.

Calibration – Second Source Standards: Second source standards shall be obtained from a different **manufacturer** than the original standard, unless one is not available. The manufacturer refers to the producer of the standard, not the vendor. The requirement for a second source standard for the initial calibration verification is waived if a second source standard is used for the continuing calibration verification. See DoD Clarification Box #37. Deviations from this requirement require project-specific approval from appropriate DoD personnel (e.g., Project Manager, Quality Assurance Officer).

The ~~freshness~~date of preparation of each standard shall be considered when evaluating its suitability for use – this consideration shall include an assessment of the stability of the standard solution, as well as its degradation rate.

The concentration of the second source standard shall be at or near the middle of the calibration range. Criteria for the acceptance of second source verification standard results shall be established. Values chosen should be at least as stringent as those established for the continuing instrument calibration verification. The initial calibration verification shall be successfully completed prior to running any samples.

Applicable only when method-specific guidance does not exist.

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e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference.

Calibration – Initial Calibration Points: Criteria for the acceptance of an initial instrument calibration must be established (e.g., correlation coefficient, **relative standard deviation**, etc.).

Exclusion of initial calibration points without technical justification is not allowed.

For example, in establishing an initial calibration curve, the calibration points used shall be a contiguous subset of the original set. In addition, the minimum linearity of the curve shall either be determined by a linear regression correlation coefficient greater than or equal to 0.995 or a maximum mean percent Relative Standard Deviation (%RSD) of 20% (with no individual analyte greater than 30%).

Deviations from the above, including for problem compounds, are permitted with the approval of DoD personnel (e.g., Project Manager, Quality Assurance Officer). See DoD Clarification Box #33 for guidance on the number of points required for a calibration curve.

Applicable only when method-specific guidance does not exist.

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f) Results of samples not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit.

Calibration – Quantitative Values in a Calibration Curve:

The range of the accepted initial calibration curve reflects the quantitation range of the samples (i.e., only those sample results with concentrations contained within the range of the calibration curve are considered to be quantitative). Any data reported outside the calibration range shall be qualified as an estimated value (i.e., by a data qualifier "flag") and explained in the case narrative.

When sample concentrations **exceed** the upper limit of the calibration curve (i.e., upper quantitation limit), samples shall be diluted and reanalyzed (if possible) to bring them within the calibration curve. When sample concentrations **fall below** the lower limit of the calibration curve (i.e., below the lower quantitation limit), then either the method shall be modified (e.g., initial calibration re-run, thereby re-establishing the potential range of quantitative values), or the resulting data shall be qualified as having estimated values.

The laboratory's **reporting limit** shall lie within the calibration range, at or above the lower quantitation limit. If the client **requires** a reporting limit that lies **below** the lower limit of the calibration curve (i.e., below the quantitation limit), then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the reporting limit shall be no lower than a low level check standard, designed to verify the integrity of the curve at the lower limits.

See also DoD Clarification Box D-10 addressing Detection Limits, as well as Definitions for Quantitation Limit and Reporting Limit.

Applicable only when method-specific guidance does not exist.

3~~1~~3

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed. Data associated with an unacceptable initial instrument calibration shall not be reported.

h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits).

Calibration Standards – Laboratory Involvement: DoD recognizes that achievability of these limits/levels by the required method is a key variable. To avoid conflicts related to this issue, DoD expects laboratory involvement (government or private) during the planning phase of the project (QAPP preparation) to ensure proper selection of methods and instrumentation. If the proposed laboratory for the project work is unavailable for this consultation (e.g., not yet selected), a government laboratory may be consulted to establish these parameters. This early involvement of a laboratory is integral in ensuring efficient planning and implementation of the project.

Applicable to all methods.

3~~2~~4

i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard. The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

Calibration – Initial Calibration: In completing work for DoD, ~~when the number of calibration points is not specified by the method,~~ the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. All reported target analytes and surrogates shall be included in the initial calibration. For multi-component analytes, such as PCBs, toxaphere, and dioxins/furans, a separate initial calibration may be required. See DoD Clarification Box #30 in Section 9.4.2.1.e for additional implementation requirements pertaining to this subject.

Applicable when method-specific guidance does not exist.

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9.4.2.2 *Continuing Instrument Calibration Verification*

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

Calibration – Continuing Instrument Calibration Verification: ~~The DoD Implementation Clarifications included in Section 9.4.2 are only applicable when method-specific guidance does not exist.~~ The validity of the initial calibration shall be verified prior to sample analyses by an **acceptable** continuing instrument calibration verification with each analytical batch. As long as the continuing calibration verification (CCV) is acceptable, a new initial instrument calibration is not necessary.

Applicable when method-specific guidance does not exist.

3~~4~~6

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.

Calibration – Continuing Calibration Verification Frequency: At least one of the ~~continuing calibration verification (~~CCV~~)~~ standards shall fall below the middle of the calibration range. At a minimum, additional periodic CCVs shall be run whenever required by the applicable method. When the methods specify that CCVs shall be run at specific sample intervals (e.g., every 10 samples), the count of these samples shall ~~include all QC~~ be of field samples only~~(i.e., each injection is considered to be a sample)~~. However, QC samples must be run with their associated batch. The grouping of QC samples from a variety of batches is not an acceptable practice. If the method does not specify an interval ~~at which~~for periodic CCVs ~~shall be completed, they shall~~, at a minimum, bracket every preparatory batch (i.e., at least every 20 field samples). More frequent CCVs are recommended for more difficult matrices.

Applicable to all methods.

3~~5~~7

c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

Calibration (CCV) – Raw Data Records: Raw records shall also include the analyst's name.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, datea, and signature/initials of person performing manual operation.

Applicable to all methods.

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d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

Calibration – CCV Criteria:

- The source of the standard(s) for analysis shallcan be the standard(s) used for the initial calibration or standard(s) from another source.
- All reportedreportable target analytes applicable to the method shall be included in the CCV. Where multi-component, multi-analyte tests are being performed, a single multi-component continuing calibration is acceptable.
- The baseline for comparison for the CCV is the initial calibration (and the original standards). Specific criteria for evaluation of success or failure of the CCV may include: percent difference/drift from the RSD established for the initial calibration, minimum response factor checks, and confirmation that the retention time is within an acceptable window. For DoD, the %RSDpercent drift/percent difference of the CCV standard shall be less than 15% of the initial calibration for organic methods and less than 10% of the initial calibration for inorganic methods or shall be equivalent to the percent drift the standard method would have allowed. If the mean value for all target analytes is used, no percent drift for an individual analyte shall exceed 25%.

Applicable when method-specific guidance does not exist.

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e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory shall demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new instrument calibration must be performed. If the laboratory has not demonstrated successful performance, additional sample analyses shall not occur until a new initial calibration curve is established and verified.

Calibration – Reporting Data from Noncompliant CCV: If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of two consecutive CCVs, then the concentrations of the two CCVs must be at two different levels within the original calibration curve. As stated in DoD Clarification Box #35, at least one of the CCV standards shall fall below the middle of the initial calibration range.

Applicable to all methods.

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However, sample data associated with an unacceptable calibration verification check may be reported as qualified data under the following special conditions:

i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, these sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

Calibration – ~~J~~Q Flag Reporting for Noncompliant CCV: Project-specific permission from appropriate DoD personnel is required to report data generated from the initial run with the noncompliant CCV. If this permission is granted, and these data are reported, they shall be qualified through the use of a "~~J~~Q" flag, and explained in the case narrative.

Applicable to all methods.

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10.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

10.1 Methods Documentation

a) The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples, and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.

b) All instructions, standards, manuals, and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

10.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain standard operating procedures (SOPs) that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer or internally written documents.

b) The test methods may be copies of published methods as long as any changes in the methods are documented and included in the methods manual. (See Section 10.1.2.)

SOPs - Requirements: Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

SOPs must document complete laboratory-specific instructions regarding equipment, processes, and procedures to a level of detail that would allow a technically qualified individual to repeat the procedure.

~~Each SOP shall provide sufficient detail such that a technically qualified analyst can perform the analysis without reference to other documents.~~ While published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP as listed in Section 10.1.2.b) Items 1-23, it is anticipated that additional information beyond the published test method documentation shall be required.

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c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized.

e) Each SOP shall clearly indicate the effective date of the document, the revision number, and the signature(s) of the approving authority.

SOPs – Archiving of SOPs: All SOPs shall be archived for historical reference in accordance with Section 12.1 (Record Keeping Systems).

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10.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

SOPs – Modifications to Existing Methods: Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

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b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

~~**DoD Implementation Clarification**~~**SOPs – Analytical Method SOPs:** These requirements apply to all Analytical Method SOPs. While published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP, as listed immediately below, it is anticipated that additional information beyond the published test method documentation will be required, including, but not limited to:

- Troubleshooting;
- Personnel qualifications;
- Data management and records; and
- Computer hardware and software.

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1) Identification of the test method;
2) Applicable matrix or matrices;
3) Method detection limit;
4) Scope and application, including components to be analyzed;
5) Summary of the test method;
6) Definitions;
7) Interferences;
8) Safety;
9) Equipment and supplies;
10) Reagents and standards;
11) Sample collection, preservation, shipment, and storage;
12) Quality control;
13) Calibration and standardization;
14) Procedure;
15) Calculations;
16) Method performance;
17) Pollution prevention;
18) Data assessment and acceptance criteria for quality control measures;
19) Corrective actions for out-of-control data;
20) Contingencies for handling out-of-control or unacceptable data;
21) Waste management;
22) References; and
23) Any tables, diagrams, flowcharts, and validation data.

10.2 Test Methods

a) The laboratory shall use appropriate test methods and procedures for all tests and related activities within its responsibility (including sample collection, sample handling, transport and storage, sample preparation, and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

1) When the use of specific test methods for a sample analysis is mandated or requested, only those methods shall be used.

2) Where test methods are employed that are not required, as in the Performance-Based Measurement System (PBMS) approach, the methods shall be fully documented and validated, and be available to the client and other recipients of the relevant reports. (See Section 10.2.1 and Appendix C).

10.2.1 Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required (See Appendix C and Section 6.2). In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that would impact the results of a specific test method), e.g., water, solids, biological tissue, and air. In addition, for analytes that do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

Capability – New Methods Capability: In the case where the laboratory is introducing a new method, demonstration of performance shall be determined using an external source of information, when available (e.g., the published method~~, Standards, or certified reference materials~~). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not "benchmark against itself" by using internal comparisons to initial runs to demonstrate capability.

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b) Thereafter, continuing demonstration of method performance, as per the quality control requirements in Appendix D, (such as laboratory control samples) is required.

Capability – Method Sensitivity Checks: The initial and continuing demonstration of capability shall include verification of method sensitivity checks (e.g., through the use of quarterly method detection verification) and demonstrated measurements of accuracy and precision (e.g., such as the production and review of quality control charts). These requirements apply to each matrix of concern.

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c) In all cases, the appropriate forms, such as the Certification Statement (See Appendix C), must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement must be retained by the laboratory.

d) A demonstration of capability must be completed each time there is a significant change in instrument type, personnel, or test method.

Capability – Significant Change: "Significant change" always refers to any change in personnel. In addition, it includes any change in instrumentation or in test methods that potentially impacts the precision, ~~and~~ accuracy, sensitivity, and selectivity of the output (e.g., a change in the detector, column, matrix, or other components of the sample analytical system, or a method revision). Requirements for meeting a "Demonstration of Capability" are further addressed in Appendix C.

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e) In laboratories with a specialized work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

f) When a work cell(s) is employed, and the members of the cell change, the new employee(s) must work with experienced analysts in the specialty area and this new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the 4 preparation batches following the change in personnel must not result in the failure of any batch acceptance, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must repeat the demonstration of capability (Appendix C).

g) When a work cell(s) is employed, the performance of the group must be linked to the training record of the individual members of the work cell (See Section 6.2).

Work Cell – Definition of Work Cell: A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation/extraction and analysis. To ensure that the entire preparation-extraction-analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the Demonstration of Capability at each individual step in the sequence as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts that performs the same step in the same process (e.g., extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

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10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative sub-samples.

Sampling – Deviations from Laboratory's Sampling Procedures: Sampling procedures shall also address laboratory practices for the handling, sub-sampling, and documenting of extraneous materials (e.g., rocks, twigs, vegetation) present in samples. The handling of multi-phase samples shall be addressed in specific sampling procedures as appropriate. When a client requires deviations from the laboratory's documented sampling procedure, all deviations shall be recorded in detail in laboratory records and in all test reports. Additionally, the laboratory shall use recognized consensus standards (e.g., ASTM standards) where available for these procedures.

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10.4 Data Verification

Calculations and data transfers shall be subject to appropriate checks.

a) The laboratory shall establish SOPs to ensure that the reported data are free from transcription and calculation errors.

b) The laboratory shall establish SOPs to ensure that all quality control measures are reviewed and evaluated before data are reported.

Data – Data Verification Procedures: Data verification (review) shall consist of at least the following procedures:

1. Determinations of whether the results of testing, examining, or analyzing the sample meet the laboratory's requirements for interpretation, precision and accuracy.
2. Checks to determine accuracy of calculations, conversions, and data transfers.
3. Checks for transcription errors, omissions, and mistakes.
4. Checks to determine consistency with project-specific ~~data~~measurement quality objectives (~~D~~MQOs).
5. Checks to ensure that the appropriate preparatory and analytical SOPs and standardized methods were followed, and that Chain-of-Custody (COC) and holding time requirements were met.
6. Checks to ensure that requirements for calibration and calibration verification standards were met, and that QC samples (e.g., method blanks, LCSs) met criteria for precision, accuracy, and sensitivity.

~~7.Procedures for verifying the reliability of the test or analytical results shall be explained to include descriptions of programmed self-protection, self-correction, or warning measures, if the laboratory uses an electronic data processor.~~

~~8.~~7. The case narrative shall accurately explain any anomalous results and any corrective actions taken, and all data flags shall be checked to ensure appropriate and accurate use.

~~9~~8. A tiered or sequential system of verification, consisting of at least three levels with each successive check performed by a different person. This three-tier approach should include (at a minimum): 100% review by the analyst, 100% verification review by a technically qualified supervisor or data review specialist, and a final administrative review. The final administrative review will verify that previous reviews were documented properly and that the data package is complete.

Additionally, as part of its internal quality assurance program, the Quality Assurance Officer or designee, shall review at a minimum, 10% of all data packages for technical completeness and accuracy. This review is part of the oversight program and does not have to be completed in "real time."

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10.5 Documentation and Labeling of Standards and Reagents

Documented procedures shall exist for the purchase, reception, and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used, unless it is verified by the laboratory.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) Records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date, and preparer's initials.

Documentation – Lot Number: The records shall include appropriate lot numbers for the standard.

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d) All containers of prepared reagents and standards must bear a unique identifier and expiration date and be linked to the documentation requirements in Section 10.5.c) above.

10.6 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage, or retrieval of test data, the laboratory shall ensure that:

Electronic Data – Audit Trails: The following applies to audit trails, as well as test data.

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a) All requirements of this Standard (i.e., NELAP Chapter 5) are complied with. Sections 8.1 through 8.11 of the EPA Document "2185 - Good Automated Laboratory Practices" (1995), shall be adopted as the standard for all laboratories employing microprocessors, computers, as well as, laboratories employing Laboratory Information Management Systems.

b) Computer software is documented and adequate for use.

c) Procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission, and data processing.

Data – Automated Processes: At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime new software is purchased or the programming code is modified or otherwise manipulated, and applies even in cases where commercial software is used as part of the process.

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d) Computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data.

e) It establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

11.0 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY, AND SAMPLE RECEIPT

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

11.1 Sample Tracking

a) The laboratory shall have a documented system for uniquely identifying the items to be tested to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size, or other physical characteristic, such as amber glass or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration. (See Section 11.3.d.)

e) In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

11.2 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples that do not meet the following criteria must be flagged in an unambiguous manner, clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

Sampling – Sample Acceptance: The laboratory shall have procedures documented in the Quality Manual or related documentation (as discussed in Sections 5.2.i. and 5.2.k.) which address methods by which the laboratory confirms that it has the capability ~~and capacity~~ to accept new samples before such acceptance occurs. The laboratory shall also follow any additional method specific requirements concerning sample acceptance.

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a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type, and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers;

d) Adherence to specified holding times;

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) Procedures to be used when samples show signs of damage or contamination.

11.3 Sample Receipt Protocols

a) Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, shall be recorded. All items specified in Section 11.2 above shall be checked.

   1) All samples that require thermal preservation shall be considered acceptable if the arrival temperature is either within +/-2° C of the required temperature or the method specified range. For samples with a specified temperature of 4° C, samples with a temperature ranging from just above the freezing temperature of water to 6° C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criterion. In these cases, the samples shall be considered acceptable, if there is evidence that the chilling process has begun, such as arrival on ice.

Sampling – Temperature Measurements: The temperature measurement shall be verified through the use of a temperature blank (for each transport container [e.g., cooler]) or other measurement when a temperature blank is not available (e.g., IR gun) when applicable.

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2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

Sampling – Chemical Preservation of Samples: This shall also be performed when the continued preservation of the sample is in question (due to sample interaction with the preservative); as applicable to samples that cannot be checked upon receipt (e.g., VOCs); and/or for samples whose preservative may have deteriorated for any other reason.

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b) The results of all checks shall be recorded.

c) Where there is any doubt as to the item's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory should consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample receipt acceptance criteria listed in Sections 11.3.a), 11.3.b), or 11.3.c), the laboratory shall either:

Sampling – Consultation ~~W~~with Client: This consultation shall be immediate and timely (i.e., by the next business day).

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1) Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

2) Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately "qualified" on the final report.

Sampling – Documentation When Acceptance Criteria Not Met: Additional guidance on this issue is provided in Section 13.a) (Laboratory Report Format and Contents).

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d) The laboratory shall utilize a permanent chronological record, such as a log book or electronic database, to document receipt of all sample containers.

Data – Electronic Databases: Use of electronic database systems shall meet the requirements specified in Section 10.6. (Computer and Electronic Data Related Requirements).

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1) This sample receipt log shall record the following:

i. Client/Project Name;

ii. Date and time of laboratory receipt;

iii. Unique laboratory ID code (See 11.1); and

iv. Signature or initials of the person making the entries.

2) During the log-in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request, and readily available to individuals who will process the sample. Note: The placement of the laboratory ID number on the sample container is not considered a permanent record.

i. The field ID code that identifies each container must be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection must be linked to the sample container and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain-of-custody (COC) record (Section 12.4), if utilized, shall be maintained.

Sampling – Legal COC and Sample Custody: Legal COC procedures, as addressed in Section 12.4, shall be required only as specified by DoD Project or Contract personnel. Standard requirements for sample custody are outlined in Sections 12.1, 12.2, and 12.3 and shall be followed as the default requirement.

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11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, contamination, or damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored, and recorded where necessary.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples that require thermal preservation shall be stored under refrigeration which is +/-2° C of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4° C, storage at a temperature above the freezing point of water to 6° C shall be acceptable.

Sampling – Refrigerated Samples: When refrigeration or freezing is required, the laboratory shall ensure that daily monitoring is performed ~~7~~5 days per week and that there shall be no break in monitoring that exceeds 60 continuous hours in any given 7-day period to ~~as~~ensure that the samples remain within an acceptable range. ~~A variety of techniques can be used to ensure that the proper temperature is continuously maintained.~~

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2) Samples shall be stored away from all standards, reagents, food, and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

Sampling – Cross-~~Concentration~~Contamination: The laboratory shall have procedures in place to ensure that cross-contamination does not occur. ~~For example, s~~Samples designated for volatile organics testing shall be segregated from other samples~~., while s~~ Samples suspected to contain high levels of volatile organics ~~should be~~shall be further isolated from other volatile organics samples or ~~. S~~storage blanks ~~may~~shall be used to verify that no cross-contamination has occurred.

~~59~~63

b) Sample fractions, extracts, leachates, and other sample preparation products shall be stored according to Section 11.4.a) above or according to specifications in the test method.

c) Where a sample or portion of the sample is to be held secure (e.g., for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

11.5 Sample Disposal

The laboratory shall have SOPs for the disposal of samples, digestates, leachates, and extracts or other sample preparation products.

Sampling – Disposal Records: The laboratory shall maintain appropriate documentation and records demonstrating that samples have been properly disposed, in accordance with Federal, State, and local regulations.

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12.0 RECORDS

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records that document all laboratory activities. The laboratory shall retain on record all original observations, calculations and derived data, calibration records, and a copy of the test report for a minimum of 5 years.

There are two levels of record keeping: (1) sample custody or tracking and (2) legal or evidentiary chain-of-custody. All essential requirements for sample custody are outlined in Sections 12.1, 12.2, and 12.3. The basic requirements for legal chain-of-custody (if required or implemented) are specified in Section 12.4.

Sampling – Legal COC and Sample Custody: Legal COC procedures, as addressed in Section 12.4, shall be required only as specified by DoD Project or Contract personnel. Standard requirements for sample custody are outlined in Sections 12.1, 12.2, and 12.3 and shall be followed as the default requirement.

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12.1 Record Keeping System and Design

The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, preparation, calibration, or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification, shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

d) All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by,” “prepared by,” or “reviewed by.”

e) All generated data, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files, or markings. All corrections to record keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to Section 10.6 for Computers and Electronic Data-Related Requirements.

12.2 Records Management and Storage

a) All records (including those pertaining to calibration and test equipment), certificates, and reports shall be safely stored, and held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in Sections 12.3 and 12.4, shall be retained for a minimum of five years from last use. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

c) Records stored or generated by computers or personal computers (PCS) shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage, and reporting.

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions in the event that a laboratory transfers ownership or goes out of business. (See NELAP Section 4.1.8.e.)

12.3 Laboratory Sample Tracking

12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) Sample preservation, including appropriateness of sample container and compliance with holding time requirement;

b) Sample identification, receipt, acceptance or rejection, and log-in;

c) Sample storage and tracking, including shipping receipts, transmittal forms, and internal routing and assignment records;

d) Sample preparation, including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, and reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions, and preventative maintenance;

h) Calibration criteria and frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment, and reporting conventions;

j) Method performance criteria, including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) All automated sample handling systems; and

n) The laboratory shall have documented procedures for the receipt, retention or safe disposal of calibration or test items, including all provisions necessary to protect the integrity of the laboratory.

12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples, and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) A written description or reference to the specific test method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) Copies of final reports;

d) Archived standard operating procedures;

e) Correspondence relating to laboratory activities for a specific project;

f) All corrective action reports, audits, and audit responses;

g) Proficiency test results and raw data; and

h) Data review and cross checking.

12.3.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) Laboratory sample ID code;

b) Date and time of analysis;

c) Instrumentation identification and instrument operating conditions/parameters (or reference to such data);

d) Analysis type;

e) All manual calculations; and

f) Analyst's or operator's initials/signature.

12.3.4 Administrative Records

The following shall be maintained:

a) Personnel qualifications, experience, and training records;

b) Records of demonstration of capability for each analyst; and

c) A log of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory record.

12.4 Legal/Evidentiary Custody

The use of legal COC protocols may be required by some State or Federal programs. In addition to the records listed in Section 12.3 and the performance standards outlined in Sections 12.1 and 12.2, the following protocols shall be incorporated if legal COC is implemented by the organization.

Sampling – Legal COC Protocols: The requirements for legal COC, as specified in Section 12.4, shall be required only when specified by DoD Project or Contract personnel. In all other cases, the standard requirements for sample custody, as outlined in Sections 12.1, 12.2, and 12.3, shall be followed and documented.

Legal COC begins at sample collection, unless otherwise specified by the applicable regulatory program. Legal COC ends after laboratory analysis of the sample is completed, at the point when the sample, sample aliquot, and sample extracts/digestates are disposed of. In all cases, laboratory disposal procedures shall be in accordance with Section 11.5 (Sample Disposal).

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12.4.1 Basic Requirements

The legal COC records shall establish an intact, continuous record of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) A sample is in someone's custody if:

   1) It is in one's actual physical possession.

   2) It is in one's view, after being in one's physical possession.

   3) It is in one's physical possession and then locked up so that no one can tamper with it.

   4) It is kept in a secured area, restricted to authorized personnel only.

b) The COC records shall account for all time periods associated with the samples.

c) The COC records shall identify individuals who physically handled individual samples.

d) In order to simplify record keeping, the number of people who physically handle the sample should be minimized. A designated sample custodian, who is responsible for receiving, storing, and distributing samples, is recommended.

e) The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

f) Legal COC shall begin at the point established by the Federal or State oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

g) The COC forms shall remain with the samples during transport or shipment.

h) If shipping containers and/or individual sample containers are submitted with sample custody seals and any seals are not intact, the lab shall note this on the COC.

i) Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent COC documentation.

j) Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed or the sample is disposed.

12.4.2 Required Information in Custody Records

In addition to the information specified in Sections 11.1.a) and 11.1.b), tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling procedure;

b) Signatures of all personnel who physically handle the sample(s);

c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis, and reporting; and

d) Common carrier documents.

12.4.3 Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

a) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.

b) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).

c) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

d) Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian or other authority.

12.4.4 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates, or extracts to another party are subject to all of the requirements for legal COC.

12.4.5 Sample Disposal

a) If the sample is part of litigation, disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user, and/or submitter of the sample.

b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

c) Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.

13.0 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously, and objectively. The results shall normally be reported in a test report and shall include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats, such as monthly operating reports, may not require all items listed below; however, the laboratory shall provide all the required information to its client for use in preparing such regulatory reports.

Reporting Requirements: The reporting requirements for work produced for DoD are outlined in Appendix DoD-A. This appendix follows all the NELAP appendices. [Pending]

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a) Except as discussed in Section 13.b), each report to an outside client shall include at least the following information (those prefaced with “where relevant” are not mandatory):

1) A title (e.g., "Test Report," or "Test Certificate," "Certificate of Results," or "Laboratory Results”);

2) Name and address of laboratory, and location where the test was carried out if different from the address of the laboratory, and phone number with name of contact person for questions;

3) Unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (e.g., 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report and that the report contains a specified number of pages.

4) Name and address of client, where appropriate, and project name, if applicable;

5) Description and unambiguous identification of the tested sample, including the client identification code;

6) Identification of test results derived from any sample that did not meet NELAC sample acceptance requirements, such as improper container, holding time, or temperature;

7) Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis, if the required holding time for either activity is less than or equal to 48 hours;

8) Identification of the test method used or unambiguous description of any nonstandard method used;

9) If the laboratory collected the sample, reference to sampling procedure;

10) Any deviations from (such as failed quality control), additions to, or exclusions from the test method (such as environmental conditions), and any nonstandard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.

11) Measurements, examinations, and derived results supported by tables, graphs, sketches, and photographs, as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as grams per liter (g/L) or milligrams per kilogram (mg/kg); and for Whole Effluent Toxicity, identify the statistical package used to provide data;

12) When required, a statement of the estimated uncertainty of the test result;

13) A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;

14) At the laboratory's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) At the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

16) Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.; and

17) Clear identification of numerical results with values outside of quantitation levels.

b) Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in 1 through 17 above readily available for review by the accrediting authority. However, formal reports detailing the information are not required if:

1) The in-house laboratory is itself responsible for preparing the regulatory reports; or

2) The laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

c) Where the certificate or report contains results of tests performed by subcontractors, these results shall be clearly identified by subcontractor name or applicable accreditation number.

d) After issuance of the report, the laboratory report shall remain unchanged. Material amendments to a calibration certificate, test report, or test certificate after issue shall be made only in the form of a further document or data transfer, including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments shall meet all the relevant requirements of this Standard.

e) The laboratory shall notify clients promptly, in writing, of any event, such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate, or amendment to a report or certificate.

f) The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile, or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.

g) Laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

Quality Manual – Supplemental Manuals: As noted in the DoD Introduction to this document, DoD plans to supplement this Manual with other standardized documents and formats to support and unify the laboratory analysis and reporting process. It is anticipated that a standardized Laboratory Report format will be issued as part of this continuing effort. In the meantime, there may be additional component-specific or project-specific requirements that supplement those listed above.

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14.0 SUBCONTRACTING ANALYTICAL SAMPLES

a) The laboratory shall advise the client in writing of its intention to subcontract any portion of the testing to another party.

b) Where a laboratory subcontracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed.

c) The laboratory shall retain records demonstrating that the above requirements have been met.

15.0 OUTSIDE SUPPORT SERVICES AND SUPPLIES

a) Where the laboratory procures outside services and supplies other than those referred to in this Standard in support of tests, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests.

b) Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials, and services comply with specified requirements. The laboratory should, wherever possible, ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.

Materials Handling: The laboratory **shall** ensure that materials are inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.

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c) The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

Supplier Records: These records shall include date of receipt, expiration date (where applicable), source (i.e., provider or supplier), lot number, and calibration and verification records and certifications for whatever supplies and services may impact the usability of associated test results. Examples of these materials that may have an impact on the quality of data include: solvents, standards, and Class A glassware, and sample containers. Furthermore, all of these supplies shall be maintained according to the applicable requirements specified in Sections 9.3 and 10.5.

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16.0 COMPLAINTS

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with Section 3.1. Records of the complaint and subsequent actions shall be maintained.

Complaints/Problems Response System: The laboratory's Quality System shall contain a process for responding to complaints and/or problems. At a minimum, this will include tracking of quality checks, internal audits, and quality control trending. Documentation of this response and resolution of the problem, as applicable to DoD, shall be maintained. In addition, the laboratory ~~is expected to~~shall use this information as part of its Quality System to identify patterns of problems and to correct them. These logs shall be available for DoD review, to help DoD assess the effectiveness of the laboratory's corrective action process. This information will be considered to be confidential, but will, nonetheless, be used by DoD to assess the effectiveness of the laboratory's quality system.

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NELAC APPENDICES

APPENDIX A - REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2

American Association for Laboratory Accreditation April 1996. General Requirements for Accreditation

"American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)", 1994

Catalog of Bacteria, American Type Culture Collection, Rockville, MD

EPA 2185 - Good Automated Laboratory Practices, 1995 available at www.epa.gov/docs/etsdwe1/irm_galp/

"Glossary of Quality Assurance Terms and Acronyms", Quality Assurance Division, Office of Research and Development, USEPA

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods", September 30, 1994, Second draft.

International vocabulary of basic and general terms in metrology (VIM): 1984. Issued by BIPM. IEC. ISO. and OIML

ISO Guide 3534-1: "Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms"

ISO Guide 7218: Microbiology - General Guidance for Microbiological Examinations

ISO Guide 8402: 1986. Quality - Vocabulary

ISO Guide 9000: 1994 Quality management and quality assurance standards - Guidelines for selection and use

ISO Guide 9001: 1994 Quality Systems - Model for quality assurance in design/development, production, installation and servicing

ISO Guide 9002: 1994 Quality systems - Model for quality assurance in production and installation

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories

"Laboratory Biosafety Manual", World Health Organization, Geneva, 1983

Manual for the Certification of Laboratories Analyzing Drinking Water Revision 4, EPA 815-B-97-001

Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, 1981

NELAC Standards, Chapters 1-6. Revision 12. July 1, 1999.

Performance Based Measurement System, EPA EMMC Method Panel, PBMS Workgroup, 1996

APPENDIX B - DEFINITIONS FOR QUALITY SYSTEMS

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

Quality Systems Definitions: The Quality Systems Committee is the group appointed by NELAC that created and continues to modify NELAP Chapter 5 (Quality Systems). Terms not included in the NELAC Glossary, but defined by DoD, are included in gray text boxes throughout this Appendix.

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Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [NELAC Section 5.2.3]

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Aliquot – A discrete, measured, representative portion of sample taken for analysis. (Source: TEAM, EPA QAD Glossary)

Analysis Duplicate: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (Source: EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Detection Limit: The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (Applicable only to radiochemistry)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (Quality Systems)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria.

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples.

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample: A subsample for analysis with a composition known to the submitter. The analyst/ laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibrate: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration: The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring device, or the correct value of each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response. (NELAC)

Calibration Method: A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody: An unbroken trail of accountability that ensures the physical security of samples, and includes the signatures of all who handled the samples.

Chemical -- Any element, compound or mixture of elements and/or compounds. Frequently, chemical ~~A~~ substances ~~that is~~ classified by the CAS rules of nomenclature for the purposes of identification for a hazard evaluation. (Source: OSHA Glossary)

Client -- The party that has agreed to pay the bill for services rendered by the laboratory, and with whom the laboratory has a contractual relationship for that project. For a laboratory, this is typically the prime contractor who originally hires the laboratory for the project, and who signs the contract as the receiver of services and resulting data. In cases where the laboratory has a direct contractual relationship with DoD, the client shall be the government's authorized technical representative. It is understood that typically

other "clients" are present at other levels of the project, but they may be removed from the day-to-day decisionmaking (e.g., installation representatives, service center representatives, various other government officials). Specific circumstances may require the direct notification of these other clients, in addition to the prime contractor or DoD representative; these circumstances shall be included as part of specific project requirements. (Source Team)

Compound -- A unique combination of chemical elements, existing in combination to form a single chemical entity. (Source: Team)

Component – A single chemical entity, such as an element or compound. Multiple components may comprise one analyte. (Source: OSHA Glossary, Team)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified. (NELAC)

Confirmation: Verification of the presence/identity of a component that may include (NELAC):

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors;
- Additional cleanup procedures, or;
- Alternative technique or conditions.

Conformance: An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ ASQC E4-1994)

Consensus Standards – A protocol established by a recognized authority (e.g., American Society for Testing and Materials [ASTM], American National Standards Institute [ANSI], or the Institute for Electrical and Electronic Engineers [IEEE]).

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.) (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form. (EPA-QAD)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Definitive Data – Data that are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data in the form of paper print-outs or electronic files. Data shall satisfy QA/QC requirements. For data to be definitive, either analytical or total measurement error shall be determined and documented. (Source: Data Quality Objectives Process for Superfund)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Desorption Efficiency: The mass of target analyte recovered from sampling media, usually a sorbent tube, divided by the mass of target analyte spiked on to the sampling media expressed as a percentage. Sample target analyte masses are usually adjusted for the desorption efficiency. (NELAC)

Detection Limit: The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. See Method Detection Limit, Quantitation Limit, and Limit of Detection. (NELAC)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA- QAD)

Environmental program – An organized effort that assesses environmental concerns and leads to the collection of data, either in the field or though laboratory analysis. (Source: Variation on EPA QAD Glossary for Terms: Environmentally related measurement, environmental sample)

~~**Fraud** – The deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable. It is also defined as an intentional gross deviation from contract-specific or method-specified analytical practices, combined with the intent to conceal the deviation.~~

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136).

Holding Times (DoD Clarification): The time elapsed from the time of sampling to the time of extraction or analysis, as appropriate.

Inspection: an activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ ASQC E4-1994)

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Instrument Blank: A clean sample (e. g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Key Staff – At a minimum, the following managerial and supervisory staff (however named): Executive Staff (e.g., Chief Executive Officer, Chief Operating Officer, Laboratory Director, Technical Director); Technical Directors/Supervisors (e.g., Section Supervisors for Organics and Inorganics); Quality Assurance Systems Directors/Supervisors (e.g., QA Officer, Quality Auditors); and Support Systems Directors/Supervisors (e.g., Information Systems Supervisor, Purchasing Director, Project Manager).

Laboratory: A body that calibrates and/or tests.

NOTES:

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.

2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing at or from a permanent location, from a temporary facility, or a mobile facility. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank or spiked blank): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit, Detection Limit, and Quantitation Limit (Analytical Chemistry, 55, p. 2217, December 1983, modified)

Manager (however named): The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix: The component or substrate that may contain~~s~~ the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or
- Saline/Estuarine source. Includes surface water, groundwater and effluents.
- Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/ Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non- aqueous liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device.

Matrix Spike (spiked sample, fortified sample): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS).

Matrix Spike Duplicate (spiked sample/ fortified sample duplicate): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS).

May: Denotes permitted action, but not required action. (NELAC)

Media: Material that supports the growth of a microbiological culture.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results. It is processed simultaneously with samples of similar matrix and under the same conditions as the samples. (NELAC).

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Must: Denotes a requirement (mandatory). (Random House College Dictionary)

National Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Nonconformance: An indication or judgement that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.

Objective Evidence: Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified. (ASQC)

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and or reagents added at the time of sample collection (or later) to maintain the chemical and or biological integrity of the sample. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC Section 2.1]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Protocol: A detailed written procedure for field and/ or laboratory operation (e. g., sampling, analysis) which must be strictly followed. (EPA- QAD)

Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance (Project) Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA- QAD)

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample: An uncontaminated sample matrix with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra- laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA- QAD)

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may also be called a Quality Assurance Plan or Quality Plan.

NOTE – The quality manual may call up other documentation relating to the laboratory's quality arrangements.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ ASQC E- 41994)

Quantitation Limits: The maximum or minimum levels, concentrations, or quantities of a target that can be quantified with the accuracy required by the intended use of the data user. (NELAC)

Quantitation Limits (DoD Clarification) – The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points ~~establish~~are established by the upper and lower limits of the calibration range.

Range: The difference between the minimum and the maximum of a set of values. (EPA- QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e. g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA- QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30- 2.1)

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC *)*

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM- 6.08)

Reference Toxicant: The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, Section 2.1). (NELAC)

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Reporting Limit – A specific concentration at or above the lower quantitation limit that is reported to the client with confidence. It is often defined on a project-specific basis. If set by the client below the lower quantitation limit, method modification is required or the client will be required to accept the lowest technically valid value which can be provided by the laboratory. For methods that only require one standard (e.g., lower limit of calibration curve is the origin), the reporting limit shall be no lower than the low level check standard.

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Sample – Portion of material collected for chemical analysis, identified by a single, unique ~~term~~alpha-numeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.

Sampling Media: Material used to collect and concentrate the target analytes(s) during air sampling

such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of nontarget substances.

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e. g., concentrations) of a variable of interest.

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI).

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI).

~~**Species** – A chemical entity that exists in a specific form (e.g., ions, molecules, solid phase compounds). (Source: Combination of multiple sources)~~

Spike: A known mass of target analyte added to a blank, sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS).

Standard Reference Material (SRM): A certified reference material produced by the U. S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method.

Supervisor (however named): The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day- to- day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/ quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/ 31/ 92).

Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA- QAD)

Technical Director: (however named) has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC Section 4.1.1.1).

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Testing Laboratory: Laboratory that performs tests. (ISO/ IEC Guide 2 - 12.4)

Test Sensitivity/Power: the minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D.2.4.). (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e. g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/ data use requirements instead of a statistical acceptance criteria (e. g. +/- 3 sigma). (ANSI N42.23- 1995, Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Tune – An injected standard required by the method as a check on instrument performance for mass spectrometry.

Validation: the process of substantiating specified performance criteria. (EPA- QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE -Verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well defined group of analysts that together perform the method analysis. The members of the group and their specific function/s within the work cell must be fully documented. (NELAC)

Sources:

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI/ ASQC E4, 1994

ANSI N42.23- 1995, Measurement and Associated Instrument Quality Assurance for Radiobioassay Laboratories

International Standards Organization (ISO) Guides 2, 30, 8402

National Institute of Standards and Technology (NIST)

National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards

Random House College Dictionary

US EPA Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

US EPA Quality Assurance Division (QAD)

40CFR Part 136

Webster's New World Dictionary of the American Language

APPENDIX C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel, or test method. (See Section 10.2.1.)

Capability – Significant Change: "Significant change" always refers to a change in personnel. In addition, it includes any change in matrix, instrumentation, or in test methods that potentially impacts the precision, ~~and~~ accuracy, sensitivity, and selectivity of the output (e.g., a change in the detector, column, or other components of the sample analytical system, or a method revision). All new analysts, regardless of experience on that instrument in another laboratory, shall complete a Demonstration of Capability.

C-1

Note: In laboratories with specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

Work Cell – Definition of Work Cell: Additional guidance on this issue is provided in Section 10.2.1.f. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation/extraction and analysis. To ensure that the entire preparation-extraction-analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the Demonstration of Capability at each individual step in the sequence as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts that performs the same step in the same process (e.g., extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

C-2

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that would impact the results of a specific test method), e.g., water, solids, biological tissue, and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the past 12 months. In addition, for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed if required by the mandatory test method or regulation. (Note: for analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. It is the responsibility of the laboratory to document that other approaches to DOC are adequate, and this shall be documented in the laboratory's Quality Manual.)

a) A quality control (QC) sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or, if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery in the appropriate reporting units (such as micrograms per liter) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory will assess performance against established and documented criteria.

Capability – New Methods Evaluation: In the case where the laboratory is introducing a new method, these criteria shall be determined using an external source of information when available (e.g., the published method~~, Standard, or certified reference material~~). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not "benchmark against itself" by using internal comparisons to initial runs to establish these criteria.

C-3

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are no established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee. (See Sections 6.3 and 12.3.4.b.)

Capability – Certification Statement: All repeated incidences of testing to meet a Demonstration of Capability shall be documented and packaged with the final Certification Statement.

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Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix: _____ (Examples: laboratory pure water, soil, air, solid, biological tissue)

Method number, SOP#, Rev #, and Analyte, or Class of Analytes or Measured Parameters:
_____ (Examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method/s, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to support these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

_____	_____	_____
Technical Director's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

APPENDIX D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the laboratory's method manual (Section 10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into its method manuals.

All QC measures shall be assessed and evaluated on an ongoing basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., Section 5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

Quality Control – Corrective Action: When quality control measures fail the acceptance criteria specified in these requirements, corrective action shall be taken. Different corrective responses may be appropriate in different situations, based upon project-specific requirements and the magnitude of the problem. Examples of corrective actions ~~that may be required~~ include:

- Determining the source of the problem,
- Notifying the client,
- Reprocessing samples,
- Using data qualifiers to "flag" data, and
- Adding commentary in laboratory reports.

D-1

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated, and measures taken to correct, minimize, or eliminate the problem, if:

   i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch, or

   ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

   Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

Method Blanks: The following paragraphs restate the requirements of Section D.1.1.a)1 above, with DoD expectations with respect to the requirement highlighted in bold.

Method Blanks - Shall be performed at a frequency of one per **preparatory** batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of **method blank** contamination shall be investigated, and measures taken to correct, minimize, or eliminate the problem~~,~~ if **the concentration exceeds one-half the ~~method~~ reporting limit. If one-half the ~~method~~ reporting limit [~~M~~RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based ~~up~~on the following criteria:**

i) The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated **preparatory** batch, or
ii) The blank contamination ~~exceeds the concentration present in the samples and~~ is greater than 1/10 of the specified regulatory limit.

Any samples associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparatory batch, except when the sample analysis resulted in a nondetect. If no sample volume remains for reprocessing, the results **shall be** reported with appropriate data qualifying codes.

D-2

b) Positive Controls

1) Laboratory Control Sample (LCS) - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen, or turbidity. The results of these samples shall be used to determine batch acceptance. NOTE: the Matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (See 2 below.)

Laboratory Control Samples (LCS): The LCS shall, as a minimum, meet limits specified in the method, if available. In addition, the laboratory shall establish its own limits, based upon in-house statistical analysis of historical LCS ~~limits~~results. The acceptability of LCS results within any preparatory batch shall be based upon these in-house limits, unless the method-specified limits are more stringent, or the client has specified limits based upon the intended use of the data.

D-3

2) Matrix Spikes (MS) - Shall be performed at a frequency of 1 in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen, or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

Matrix Spike Frequency: Matrix spikes shall be performed at a frequency of 1/20 samples per matrix type if adequate sample material is provided by the field investigation. If adequate sample material is not available then the frequency of matrix spikes shall be noted in the case narrative. Additional matrix spikes may be required by project specific needs for ~~field~~ quality control. ~~The selection of these samples is particularly critical when additional sample volumes are necessary to complete the analyses.~~

D-4

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene, and polychlorinated biphenyls [PCBs] in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes, and other client requested components. However, the laboratory shall ensure, that all reported components are used in the spike mixture within a two-year time period.

Spiking Compounds:

- The protocols above shall only be required if the test method **or project-specific requirements** do not specify the spiking compounds.
- The list of "reportable components" is specified by the project.
- For DoD, "an extremely long list of components" means greater than 50 components ~~.~~reported per method. The exception does not apply to generalized analyte lists (e.g., Appendix IX). If a percentage of the component list is used, those analytes must be representative of each chemical class covered by the test method and include any project-specific analytes of concern.
- The concentration of the matrix spike shall be at or below the midpoint of the calibration range.

D-5

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document its procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

Matrix Spike Duplicates: Each duplicate named above shall be analyzed by the same specifications as its respective matrix spike. For example, matrix spike duplicates shall be performed at a frequency of 1/20 samples per matrix type. Additional matrix spikes duplicates may be required by project specific needs. ~~The selection of these samples is particularly critical when additional sample volumes are necessary to complete the analyses.~~

D-6

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Analytical Capability - (Section 10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

Capability – Significant Change: "Significant change" refers to any change in personnel. In addition, it includes any change in instrumentation or in test methods that potentially impacts the precision and accuracy, sensitivity and selectivity of the output (e.g., a change in the detector, column, or other components of the sample analytical system, or a method revision). Requirements for meeting an "Demonstration of Capability" are further addressed in Appendix C.

D-7

b) Calibration - Calibration protocols specified in Section 9.4 shall be followed.

Calibration Protocols: Protocols in Section 9.4 shall be followed, unless method or project specific procedures and criteria are available.

D-8

c) Proficiency Test Samples - The results of such analyses (Section 4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

Proficiency Testing: Proficiency Testing is discussed further in NELAP Chapter 2. If such testing reveals inaccuracies in data generation, corrective action shall be taken in accordance with the laboratory's documented procedures. DoD shall submit its own proficiency testing samples, as it deems necessary.

D-9

D.1.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate or relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

Detection Limits: A Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, and is determined from analysis of a sample in a given matrix containing the analyte.

Requirements established in 40 CFR 136B are the baseline source of information for determining MDLs. Other published statistical methods may be appropriate as supplemental resources in determining MDLs (e.g., Hubaux and Vos studies may be appropriate for methods that do not require prep, such as GC/MS volatiles in water). The following list provides clarification and expansions upon the fundamental requirements and principles outlined in 40 CFR 136 B, and shall be followed when performing work for DoD:

- As stated in 40 CFR 136 B, MDLs shall be determined using a minimum of 7 replicates. If more than 7 replicates are processed, data cannot be excluded, unless exclusion is supported with sound, documented, technically-based justification.
- MDLs are to be calculated for each analyte~~,~~ and matrix~~, and instrument~~. If multiple instruments with identical configurations are used in the laboratory, then the laboratory ~~may~~shall conduct an MDL study on at least one of the instruments, and confirm the attainability of that MDL on all instruments by using an MDL verification check sample.
- If multiple MDL results are generated from multiple instruments with identical configurations, then the highest MDL among those may be used in reporting data from all of those instruments. If a lower MDL is reported for specific samples, then the samples must have been run on that specific instrument on which the lower MDL was generated.
- MDLs shall be generated for all applicable matrices, using, at a minimum, a purified matrix free of the analytes of interest (e.g., Ottawa sand, reagent grade water). For metals, teflon chips can be used to simulate the soil matrix.
- MDLs shall be generated for all prep and cleanup methods routinely used on samples.
- An MDL verification check shall always be performed immediately following an MDL study. DoD requires that the MDL check sample be spiked at **approximately two times** the current reported MDL.
- If an annual MDL study is not performed, MDL verification checks shall be performed **quarterly**~~, if an annual MDL study is not performed~~. If the quarterly MDL verification check fails, ~~then~~additional MDL verification checks shall be performed at a higher level to set a higher MDL or the MDL study shall be re-conducted.
- For DoD, the MDL verification check sample shall be acceptable if it~~always~~ produces a response that lies at least three times above the instrument's noise level.
- Deviations from the above are permitted with the approval of DoD personnel.

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a) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available, such as odor and temperature.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results, or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a significant change in the test method or instrument type.

d) It is essential that all processing steps of the analytical method be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

Data Reduction Procedures – Automated Processes: At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime the programming code is modified or otherwise manipulated, and applies even in cases where commercial software is used as part of the process.

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D.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with Section 9.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

SOPs – Water Quality in Method SOPs: When water quality is not specified in the method, the default water quality shall be specified in the method-specific Standard Operating Procedures (SOPs) (for example, American Society for Testing and Materials [ASTM] Type I or II) and be of known, documented, and appropriate quality.

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D.1.7 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

Retention Time Verification – Frequency and Criteria: The laboratory shall follow method-specific requirements for frequency of retention time verification and criteria for acceptance. If method specific requirements do not exist, the laboratory shall develop and document the frequency of retention time verification and the acceptance criteria for retention time windows.

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b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

Data – Data Confirmation: This requirement may be waived by the client in the case of periodic monitoring of well-characterized media, which are tested by the same laboratory. For data that are required to be confirmed, all results shall be reported as confirmed or unconfirmed. If unconfirmed data are reported, they shall be identified separately in the report, with a narrative explaining why the data were not confirmed. Evaluation criteria for the confirmation of results shall be as specified by the method ~~(e.g., SW846 8000B requires a relative percent difference [RPD] of less than 40% in order for the data to be considered "confirmed".),~~unless otherwise specified by DoD personnel. If method-specific requirements do not exist, the laboratory shall develop and document acceptance criteria for the confirmation of results.

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c) The laboratory shall document acceptance criteria for mass spectral tuning.

Mass Spectral Tuning – Acceptance Criteria: These acceptance criteria are specified by the method, unless otherwise specified by DoD personnel.

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D.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 WHOLE EFFLUENT TOXICITY TESTING

D.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the test method.

1) The laboratory must demonstrate its ability to obtain consistent results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes.

   i. An intra-laboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intralaboratory variability with a specific reference toxicant for each test method. In addition, a laboratory must produce test results that meet test acceptability criteria (such as greater than 80% survival in the control), as specified in the specific test method.

   ii. Intra-laboratory precision on an ongoing basis must be determined through the use of reference toxicant tests and plotted in quality control charts. As specified in the test methods, the control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC 50 for acute tests, over time within a laboratory.

2) The frequency of reference toxicant testing shall comply with the EPA or State permitting authority requirements.

3) The EPA test methods for EPA/600/4-91-002, EPA/600/4-91-003, and EPA/600/4-90-027F do not currently specify a particular reference toxicant and dilution series; however, if the State or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements.

4) Test Acceptability Criteria (TAC) - The test acceptability criteria (e.g., the chronic *Ceriodaphnia* test, requires 80% or greater survival and an average 15 young per female in the controls), as specified in the test method, must be achieved for both the reference toxicant and effluent test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity:

   i. The control population of *Ceriodaphnia* shall contain no more than 20% males.

   ii. An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests. (See test conditions and test acceptability criteria specified for each test method.) The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

b) Negative Control - Control, Brine Control, or Dilution Water - The standards for the use, type, and frequency of testing are specified by the test methods and by permit and shall be followed.

D.2.2 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a) above.

D.2.3 Accuracy

This principle is not applicable to Whole Effluent Toxicity.

D.2.4 Test Sensitivity

a) Test sensitivity (or test power) of the tests will depend in part on the number of replicates per concentration, the significance level selected (0.05), and the type of statistical analysis. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. Test sensitivity is the minimum significant difference (MSD) between the control and test concentration that is statistically significant. If the Dunnett's procedure is used, the MSD shall be calculated according to the formula specified by the EPA test method and reported with the test results.

b) Estimate the MSD for non-normal distribution and or heterogenous variances.

c) Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

d) The MSD shall be calculated and reported for only chronic endpoints. In addition, the calculated endpoint is typically a lethal concentration of 50% (LC 50); therefore, confidence intervals shall be reported as a measure of the precision around the point estimate value. In order to have sufficient replicates to perform a reliable MSD, such tests shall have a minimum of four replicates per treatment so that either parametric or non parametric tests can be conducted.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) The methods of data analysis and endpoints will be specified by language in the permit or, if not present in the permit, by the EPA methods manuals for Whole Effluent Toxicity.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical to cumulative percentage of test organisms demonstrating a response such as death.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in Whole Effluent Toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals, which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH, or specific conductance, shall comply with the standards outlined in Appendix D.1 above.

Typographical Correction: The above reference should read Appendix D.1.6, instead of D.1.

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D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s) and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory.

c) Instruments used for routine measurements of chemical and physical parameters such as pH, dissolved oxygen (DO), conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated and/or standardized per manufacturer's instructions and Section D.1. Temperature shall be calibrated per Section 9.4.2.1. All measurements and calibrations shall be documented.

Calibration – Chemical and Physical Parameters: Instruments used for routine measurements of chemical and physical parameters, such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, weight, and temperature shall be calibrated and/or standardized per manufacturer's instructions and Section 9.4.2.1 All measurements and calibrations shall be documented.

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d) Test temperature shall be maintained, as specified in the methods manuals. The average daily temperature of the test solutions must be maintained within 1° C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be once per 24-hour period. The test temperature for continuous flow toxicity tests shall be recorded and monitored continuously.

e) Water used for culturing and testing shall be analyzed for toxic metals and organics annually or whenever the minimum acceptability criteria for control survival, growth, or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. The method specified analytes and concentration levels shall be followed.

f) New batches of food used for culturing and testing shall be analyzed for toxic organics and metals. If food combinations or recipes are used, analyses shall be performed on the final product upon the use of new lot of any ingredient. If the concentration of total organic chlorine exceeds 0.15 microgram per gram wet weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 microgram per gram wet weight, or toxic metals exceeds 20 microgram per gram wet weight, the food must not be used.

g) Test chamber size and test solution volume shall be as specified in the methods manuals.

h) Test organisms shall be fed the quantity and type food specified in the methods manuals. They shall also be fed at the intervals specified in the test methods.

i) Light intensity shall be maintained, as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal tests, the light intensity shall be measured and recorded at the start of each test.

j) At a minimum, during chronic testing, DO and pH shall be measured daily in at least one replicate of each concentration. DO may be measured in new solutions prior to organism transfer, in old solutions after organisms transfer, or both.

k) All cultures used for testing shall be maintained, as specified in the methods manuals.

l) Age and the age range of the test organisms must be as specified in the manuals.

m) The maximum holding time (lapsed time from sample collection to first use in a test) shall not exceed 36 hours without the permission of the permitting authority.

n) All samples shall be chilled to 4° C during or immediately after collection. They shall be maintained at a temperature range from just above the freezing temperature of water to 6° C and the arrival temperature shall be no greater than 6° C. Samples that are hand delivered to the laboratory immediately after collection (i.e., within 1 hour) may not meet the laboratory temperature acceptance criteria. In these cases, the laboratory may accept the samples if there is evidence (such as arrival on ice) that the chilling process has begun.

o) Organisms obtained from an outside source must be from the same batch.

D.3 MICROBIOLOGY

These standards apply to laboratories undertaking the examination of materials, products, and substances involving microbiological analysis, recovery, or testing. The procedures involve the culture media, the test sample, and the microbial species being isolated, tested, or enumerated.

a) Microbiological testing refers to and includes the detection, isolation, enumeration, and identification of microorganisms and their metabolites, as well as sterility testing. It includes assays using microorganisms, as part of a detection system and their use for ecological testing.

b) These standards are concerned with the quality of test results and not specifically with health and safety measures. In the performance of microbiological testing, laboratories must be aware of and have SOPs that conform with local, State, and national regulatory policies for the safety and health of personnel.

D.3.1 Positive and Negative Controls

a) Negative Controls:

The laboratory shall demonstrate that the cultured samples have not been contaminated through sample handling/preparation or environmental exposure. These controls shall include sterility checks of media, blanks such as filtration blanks, bottle, and buffer blanks.

1) All blanks and uninoculated controls, specified by the test method, shall be prepared and analyzed at the frequency stated in the method.

2) A minimum of one uninoculated control shall be prepared and analyzed, unless the same equipment set is used to prepare multiple samples. In such cases, the laboratory shall prepare a series of blanks using the equipment. At least one beginning and ending control shall be prepared, with additional controls inserted after every 10 samples.

3) Analyze a known negative culture.

b) Positive Controls:

Positive controls demonstrate that the medium can support the growth of the test organism and that the medium produces the specified or expected reaction to the test organism.

1) On a monthly basis, each lot of media shall be tested with at least one pure culture of a known positive reaction and shall be included with the sample test batch.

2) If routine culturing is not part of the laboratory's testing and pre-prepared media are routinely used, strict control of the storage conditions and expiration date of media shall be maintained. A positive growth control from a known positive sample shall be run with each lot to ensure that the media support growth.

3) If the laboratory has at least one known positive result of the appropriate organism during the month, a separate positive control is not required.

D.3.2 Test Variability/Reproducibility

a) Duplicates - At least 5% of the suspected positive samples shall be duplicated. In laboratories with more than one analyst, each shall make parallel analyses on at least one positive sample per month.

Sample Duplicates – Positive Results: If a sample tests positive, repeated field sampling may be required to fulfill duplication requirements.

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b) Where possible, participation in, or organization of collaborative trails, proficiency testing, or interlaboratory comparisons, either formal or informal, must be done.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a test method for its intended purpose, the laboratory shall demonstrate and document its ability to meet acceptance criteria either specified by the method or by the EPA or State program requirements. Acceptance criteria must meet or exceed these requirements and must demonstrate that the test method provides correct/expected results with respect to specified detection capabilities, selectivity, and reproducibility.

1) Accepted (official) test methods or commercialized test kits for official test methods, or test methods from recognized national or international standard organizations, may not require a specific

validation. Laboratories are required, however, to demonstrate proficiency with the test method prior to first use. This can be achieved by simultaneous, side-by-side analysis by several analysts.

2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity and reproducibility. The differences due to the matrices must be taken into account when testing different sample types.

3) The validation of microbiological test methods shall be performed under the same conditions as those for routine sample analysis. This can be achieved by using a combination of naturally contaminated products and spiked products with results that can be statistically analyzed to demonstrate that the test meets its intended purpose.

4) All validation data shall be recorded and stored at least as long as the test method is in force, or if withdrawn from active use, for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs (interlaboratory) identified by NELAP (See Section 4.2.j or 5.3.4.)

D.3.4 Test Performance

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner. (See Section D.3.1.b.)

D.3.5 Data Reduction

a) The calculations, data reduction, and statistical interpretations specified by each test method shall be followed.

b) If the test method specifies colony counts, such as membrane filter or colony counting, then the ability of individual analysts to count colonies shall be verified at least once per month, by having two or more analysts count colonies from the same plate.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared in the laboratory from the different chemical ingredients, from commercial dehydrated powders, or may be purchased ready to use.

b) Reagents, commercial dehydrated powders, and media shall be used within the shelf-life of the product and shall be documented according to 10.5. The laboratory shall retain all manufacturer supplied “quality specification statements,” which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility check including acceptability criteria, performance checks including the organism used, their culture collection reference and acceptability criteria, date of issue of specification, or statements assuring that the relevant product batch meets the product specifications.

c) Distilled water, deionized water or reverse osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media solutions and buffers. The quality of the water shall be monitored for attributes such as pH, chlorine residual, specific conductance, or metals at the specified frequency and evaluated according to the stated standards. Records shall be maintained on all activities.

d) Media, solutions, and reagents shall be prepared, used, and stored according to a documented procedure following the manufacturer’s instructions or the test method.

e) All laboratory media shall be checked to ensure they support the growth of specific microbial cultures. In addition, selective media shall be checked to ensure they suppress the growth of nontarget organisms. Media purchased pre-prepared from the manufacturer shall be checked monthly except when the use and maintenance of pure cultures is not part of laboratory procedures. In preference to using the commonly used streak method, it is better to use a quantitative procedure, where a known (often low) number of relevant organisms are inoculated into the medium under test and the recovery evaluated.

f) Each lot of laboratory detergent shall be checked to ensure that residues from the detergent do not inhibit or promote growth of microorganisms, for example, with an inhibitory residue test.

D.3.7 Selectivity

a) All confirmation/verification tests specified by the test method shall be performed according to method protocols.

b) In order to demonstrate traceability and selectivity, laboratories shall use reference cultures of microorganisms obtained from a recognized national collection or an organization recognized by the assessor body.

1) Reference cultures may be subcultured once to provide reference stocks. Appropriate purity and biochemical checks shall be made and documented. The reference stocks shall be preserved by a technique that maintains the desired characteristics of the strains. Examples of such methods are freeze-drying, liquid nitrogen storage, and deep-freezing methods. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be refrozen and reused.

2) Working stocks shall not be sequentially cultured more than five times except when:

i. It is required by standard test methods, or

ii. Laboratories can provide documentary evidence demonstrating that there has been no loss of viability, no changes in biochemical activity, and/or no change in morphology.

3) Working stocks shall not be subcultured to replace reference stocks.

4) A scheme for handling reference cultures is included in Figure D.1.

Figure D-1. USE OF REFERENCE CULTURES (BACTERIA)

Flow Chart

Reference culture from source recognized by NELAC (usually American Type Culture Collection)

Culture once
Appropriate Purity Checks and Biochemical Tests

Reference Stocks
Retained under specific Conditions:
Freeze dried, liquid nitrogen, or deep frozen storage

Thaw/Reconstitute
Purity Checks and Biochemical Tests as Appropriate

Working Stocks
Maintained under specific conditions and storage times

Regular/Daily Quality Controls

D.3.8 Constant and Consistent Test Conditions

a) The laboratory shall devise an appropriate environmental monitoring program to indicate trends in levels of contamination appropriate to the type of testing being carried out. Acceptable background counts shall be determined, and there shall be documented procedures to deal with situations in which these limits are exceeded.

b) Walls, floors, ceilings, and work surfaces shall be nonabsorbent and easy to clean and disinfect. Wooden surfaces of fixtures and fitting shall be adequately sealed. Measures shall be taken to avoid accumulation of dust by the provision of sufficient storage space by having minimal paperwork in the laboratory and by prohibiting plants and personal possessions from the laboratory work area.

c) Temperature measurement devices;

1) Where the accuracy of temperature measurement has a direct effect on the result of the analysis, temperature measuring devices, such as liquid-in-glass thermometers, thermocouple, platinum resistance thermometers used in incubators, autoclaves, and other equipment, shall be the appropriate quality to achieve the specification in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement, and they shall be calibrated to national or international standards for temperature. (See Section 9.2.1.) Calibration shall be done at least annually.

Typographical Correction: The reference at the end of this paragraph should read Section 9.2 instead of Section 9.2.1.

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2) The stability of temperature, uniformity of temperature distribution, and time required to achieve equilibrium conditions in incubators, waterbaths, ovens, and temperature controlled rooms shall be established (e.g., position, space between and height of stacks of Petri dishes).

d) Autoclaves:

1) The performance of each autoclave shall be initially evaluated by establishing its functional properties (e.g., heat distribution characteristics with respect to typical uses). Autoclaves shall be capable of meeting specified temperature tolerances. Pressure cookers fitted only with a pressure gauge are not recommended for sterilization of media or decontamination of wastes.

2) Records of autoclave operations, including temperature and time, shall be maintained. This shall be done for every cycle. Acceptance/rejection criteria shall be established and used to evaluate the autoclave efficiency and effectiveness.

e) Volumetric equipment such as automatic dispensers, dispenser/diluters, mechanical hand pipettes, and disposal pipettes, may all be used in the microbiology laboratory. Regular checks, as outlined in Section 9.4.2.1, shall be performed and documented.

f) UV Sterilizers

1) Are to be tested quarterly for effectiveness with positives (either reference cultures or positive monitoring samples) and this is to include testing of the power output of the UV bulb.

g) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements. Mechanical timers shall be checked regularly against electronic timing devices to ensure accuracy.

D.4 RADIOCHEMICAL ANALYSIS

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation, followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g., ICP-MS or TIMS) or optical (e.g., KPA) techniques are not addressed herein.

D.4.1 Negative Controls

a) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the QC measures used to assess batch acceptance. The method blank result shall be assessed against the specific acceptance criteria [Section 10.1.2] specified in the laboratory method manual [Section 10.1.2]. When the specified method blank acceptance criteria is not met, the specified corrective action and contingencies [Sections 10.1.2] will be followed. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11].

b) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry, the method blank shall be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

c) There shall be no subtraction of the required method blank [Section D.4.1.a)] result from the sample results in the associated preparation or analytical batch. This does not preclude the application of any correction factor (e.g., instrument background, analyte presence in tracer, reagent impurities, peak overlap, calibration blank, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

d) The method blank acceptance criteria [Section 10.1.2.b)18] shall address the presumed aliquot size on which the method blank result is calculated and the manner in which the method blank result is compared to sample results of differing aliquot size.

D.4.2 Positive Controls

a) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The laboratory control sample result shall be assessed against the specific acceptance criteria [Section 10.1.2.b)18] specified in the laboratory method manual [Section 10.1.2]. When the specified laboratory control sample acceptance criteria are not met, the specified corrective action and contingencies [Section 10.1.2.a)19 and 20] will be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11.]

b) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods that do not utilize an internal standard or carrier and for which there is a physical or chemical separation process and where there is sufficient sample to do so. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The matrix spike result shall be assessed against the specific acceptance criteria [Section 10.1.2.b)18] specified in the laboratory method manual [Section 10.1.2]. When the specified matrix spike acceptance criteria are not met, the specified corrective action and contingencies [Section 10.1.2.a)19 and 20] will be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11]. The lack of sufficient sample aliquot size to perform a replicate analysis should be noted in the laboratory report.

c) The activity of the laboratory control sample and matrix spike analyte(s) shall be greater than ten times and less than 100 times the a priori detection limit.

d) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration.

e) Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. isotopic uranium: U-234, -235, and -238) only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified activity level, each shall be assessed against the specified acceptance criteria.

f) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope, the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g., americium-241), medium (e.g., cesium-137) and high (e.g., cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples, the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

D.4.3 Test Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [Section 10.1.2.b)18] specified in the laboratory method manual [Section 10.1.2]. When the specified replicate acceptance criteria are not met, the specified corrective action and contingencies [Section 10.1.2.a)19 and 20] will be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11].

D.4.4 Other Quality Control Measures

a) Tracer - For those methods that utilize a tracer (i.e. internal standard), each sample result will have an associated tracer recovery calculated and reported. The tracer recovery for each sample results shall be one of the QC measures to be used to assess the associated sample result acceptance.

The tracer recovery shall be assessed against the specific acceptance criteria [Section 10.1.2.b)18] specified in the laboratory method manual [Section 10.1.2]. When the specified tracer recovery acceptance criteria are not met, the specified corrective action and contingencies [Section 10.1.2.a)19 and 20] will be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11].

b) Carrier - For those methods that utilize a carrier (i.e. internal standard) each sample will have an associated carrier recovery calculated and reported. The carrier recovery for each sample shall be one of the QC measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [Section 10.1.2.b)18] specified in the laboratory method manual [Section 10.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [Section 10.1.2.a)19 and 20] will be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)11].

D.4.5 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability - (Section 10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Proficiency Test Samples - The results of such analysis (Section 4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data. The providers of such proficiency test samples should conform to the requirements of ANSI N42.22.

D.4.6 Radiation Measurement System Calibration

Due to the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to calibrate these systems in the day of use manner done so for some types of chemical measurement instrumentation. As well, due to the nature of some radiation measurement instrumentation calibrations, it may not be practical to calibrate in a day of use manner. In addition, the calibration of modern radiation measurement instrumentation has significant differences from chemical measurement instrumentation. This section will address those practices that are necessary for proper calibration and those requirements of Section 9.4.3 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Calibration Curves - The requirements of Sections 9.4.3.b)1 through 9.4.3.b)4 for the determination of the appropriate number of standards for initial calibration are not applicable to the performance of radiochemical methods. For those radiochemical methods that may require multiple standards for initial calibration (e.g., gas-proportional counting and liquid scintillation counting) the required number shall be addressed in the laboratory method manual [Section 10.1.2.], if not addressed in the method.

b) Calibration Curve Regression - The requirements of Section 9.4.3.c) are not necessarily applicable for all radiochemical methods. Instead, where linear regression is used to fit standard response or calibration standard results to a calibration curve, the correlation coefficient shall be determined. Where nonlinear regression is used to fit standard response or calibration standard results to a calibration curve, the correlation coefficient should be determined.

c) Calibration Range - The requirements of Section 9.4.3.d) are not applicable to the performance of radiochemical methods given the noncorrelated event nature of decay counting instrumentation.

d) Calibration Verification - The LCS may fill the requirements for the performance of an initial calibration and continuing calibration verification standard as specified in Sections 9.4.4.1 and 9.4.4.2. The calibration verification acceptance criteria shall be the same as specified for the LCS.

e) Background Calibration - Background calibration measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values are subtracted from the total measured activity in the determination of the sample activity

1) For gamma spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, background calibration measurements shall be performed on a day of use basis.

f) Calibration - Instrument calibration shall be performed with reference standards as defined in Section D.4.9.a). The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

g) The frequency of calibration shall be addressed in the laboratory method manual [Section 10.1.2.13] if not addressed in the method. A specific frequency (e.g., monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

D.4.7 Method Detection Limits

Note: To be addressed in the next Chapter 5 revision.

D.4.8 Data Reduction

a) Refer to Section 10.6, ”Computers and Electronic Data Related Requirements,” of this document.

b) Method Uncertainties - The laboratory shall have the ability to trace all sources of method uncertainties and their propagation to reported results. The ISO "Guide to the Expression of Uncertainty in Measurement" and/or the NIST Technical Note 1297 on "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results" should be used in this regard.

D.4.9 Quality of Standards and Reagents

a) The QC program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the NIST, EPA, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards should conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value.

b) All reagents used shall be analytical reagent grade or better.

D.4.10 Constant and Consistent Test Conditions

a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

b) Instrument performance checks - Instrument performance checks using appropriate check sources shall be performed on a regular basis and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the performance checks of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel. For alpha and gamma spectroscopy systems, the instrument performance checks shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a day of use basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, the performance checks for counting efficiency shall be performed on a day of use basis.

D.5 AIR TESTING

Analysis for Air Toxics shall follow the essential quality controls for chemistry outlined in Appendix D.1. For air testing, the blank, laboratory control sample, and a desorption efficiency (such as charcoal tubes) shall be used. Matrix spikes and duplicate samples shall be used when feasible.

~~DD APPENDICES~~

APPENDIX DoD-A – REPORTING REQUIREMENTS

The reporting requirements outlined below are for hard copy data reports from the laboratory. They are divided into mandatory requirements for all printed data reports, and optional requirements. Optional reporting requirements are those that may be required by a specific project, depending upon the needs of the project. The following elements are required in every report: cover sheet, table of contents, case narrative, analytical results, sample management records, and QA/QC information. Information for third party review and a performance based data package may be required depending on project specific requirements or the method being used.

1. Cover Sheet. The cover sheet shall specify the following information:

- title of report (i.e., Test Report, Test Certificate);
- name and location of laboratory (to include a point of contact, phone and facsimile numbers;
- name and location of any subcontractor laboratories, and appropriate test method performed;
- contract number;
- client name and address;
- project name and site location;
- statement of data authenticity and official signature and title of person authorizing report release; and
- amendments to previously released reports shall clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report.

2. Table of Contents. Laboratory data packages should be organized in a format that allows for easy identification and retrieval of information. An index or table of contents shall be included for this purpose.

3.Case Narrative. A case narrative shall be included in each report. The purpose of the case narrative is to:

- describe any abnormalities and deviations that may affect the analytical results; and
- summarize any issues in the data package that need to be highlighted to the data user to help them assess the useability of the data.

The case narrative shall provide:

- a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed and by which laboratories;
- a list of samples that were received but not analyzed;
- a description of extractions or analyses that are performed out of holding times;
- a definition of all data qualifiers or flags used;
- identification of deviations of any calibration standards or QC sample results from appropriate acceptance limits and a discussion of the associated corrective actions taken by the laboratory; and
- appropriate notation of any other factors that could affect the sample results (e.g., air bubbles in VOC sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature and sample pH excursions, container type or volume, etc.).

4. Analytical Results. The results for each sample shall contain the following information at a minimum. (Information need not be repeated if noted elsewhere in the data package).

- project name and site location;

- field sample ID number as written on custody form;
- laboratory sample ID number;
- matrix (soil, water, oil, etc.);
- date sample extracted or prepared;
- date sample analyzed;
- method numbers for all preparation, cleanup, and analysis procedures employed;
- analyte or parameter;
- method reporting limits adjusted for sample-specific factors (e.g., aliquot size, dilution /concentration factors, moisture content);
- method quantitation limits (low-level standard concentration);
- analytical results with correct number of significant figures;
- any data qualifiers assigned;
- concentration units;
- dilution factors;
- All reported data shall reflect any dilutions or concentrations. If neat or less diluted results are available, data from both runs should be recorded and reported; and
- percent moisture or percent solids (all soils are to be reported on a dry weight basis).

The following information is optional but may be required site specifically:

- laboratory name and location (city and state);
- sample description;
- sample preservation or condition at receipt;
- date sample collected;
- date sample received;
- method detection limits;
- sample aliquot analyzed;
- final extract volume; and
- CAS number.

5. Sample Management Records. These types of records include the documentation accompanying the samples.

- chain-of-custody records;
- shipping documents;
- records generated by the laboratory which detail the condition of the samples upon receipt at the laboratory (e.g, sample cooler receipt forms);
- telephone conversation records associated with actions taken or quality issues; and
- laboratory internal sample custody records through sample analysis, transfer and disposal.

6. QA/QC Information. The minimum internal QC data package must include:

- matrix spikes percent recovery;
- relative percent difference (RPD) of required duplicates;
- LCS percent recoveries;
- surrogate percent recoveries (organics);
- method blank results; and
- preparation, analysis and other batch numbers.

7. Information for Third-Party Review. The information listed below is required only if third-party (from outside the laboratory) data validation or verification is to be performed. This information is therefore optional and is provided only when the project specific requirements specify third party review will occur.

- calibration data from the initial calibration curve;
- initial calibration verification (ICV);

- continuing calibration verification(s) (CCV);
- performance standards analyzed in conjunction with the test method (e.g., tuning standards, degradation check standards, etc.);
- preparation, analysis and other batch numbers;
- raw data (e.g., chromatograms, mass spectrum results and ICP);
- matrix spike (MS) (if applicable) (includes spike target concentration levels, measured spike concentration and calculated recoveries);
- RPD of required duplicates (e.g., MS, LCS, field duplicates);
- method blank results;
- LCS recoveries;
- surrogate recoveries (organics);
- serial dilutions (SD) percent difference (inorganics); and
- post-digestion spikies recovery (inorganics).

In addition, the data package for third party review may include:

- method detection limit studies; and
- supporting documentation (e.g., run logs, sample preparation logs, standard preparation logs).

The data validation guidelines for performance-based methods established in other DoD guidance on data review and data validation, EPA national functional guidelines, EPA regional functional guidelines, and project-specific guidelines for validation may all have distinct reporting formats. The appropriate validation guidelines should be consulted to determine what type of data package is required.

8. Performance Based Data Package. The requirements for the ***Performance based data package are the same as those defined within the definitive data package within the addition of the following items: (1) all appropriate project action level(s) and DQOs, and (2) appropriate preparatory and analysis logs.*** Refer to other DoD guidance on the Data Review of Performance Based Methods for further details on this data package.